

1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 3

AIR EMISSION SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

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G. C. RONAN, DIRECTOR
Laboratory Services Branch
Ministry of the Environment

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1986

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SECTION 3

AIR EMISSION SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

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Inorganic Trace Contaminants Section
Laboratory Services Branch
Ministry of the Environment

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INORGANIC TRACE CONTAMINANTS SECTION

SUMMARY

I. Introduction

The Inorganic Trace Contaminants Section of the Ministry of the Environment, Laboratory Services Branch is responsible for the analysis of a wide variety of sample types for metals and non-metals. The use of sensitive instrumentation and methodologies appropriate to the sample matrix, combined with quality assurance programs, ensures that the Section is able to maintain a high standard of analytical performance. This performance is monitored through regular internal quality control and assurance programs as well as participation in interlaboratory round-robins. This QA report summarizes the methodologies used for analysis of these samples and the supporting internal quality assurance data.

This report is assembled in sections that reflect the analyses performed on different sample matrices in support of the programs of the Ministry of the Environment. Coincidentally, these divisions also reflect the supervisory responsibilities within the Section.

II. Quality Control and Assurance

The objectives of the quality control and assurance programs are to ensure that all of the components of the analytical process are under control and to ensure immediate detection and correction of unacceptable analytical performance. The program monitors all of the reagents, instrumentation, calibration and recovery components of the analytical system.

A. Quality Control

Quality control of the analytical process takes place at the instrument level and is intended to ensure that the instrumentation is operating according to established criteria. This control function ensures that instrument calibration, standardization, slope and intercept, and instrumental drift meet these criteria.

B. Quality Assurance

Quality assurance of the analytical process takes place after the results have been generated and is intended to ensure that the analytical protocols of sample preparation and digestion have been carried out correctly. This control function ensures that reagent blanks, digested standards, sample duplicates and recovery materials meet established response criteria.

III. Report Format

The report consists of one page method summaries and one page data summaries of blanks, between-run controls and within-run duplicates in formats that are common to all of the parameter/matrix combinations. The method summaries give a brief outline of the sample preparation and measurement procedures. The data summaries consist of annual mean values with standard deviations.

For the within-run duplicates, the data set is subdivided into ranges approximating 0 to 20 %, 20 to 50 % and 50 to 100% of the analytical range. All results for duplicates reported to the data base that are "<" or that have been diluted into the range are excluded from the statistical analysis.

The standard deviations for blanks and between-run controls are calculated using formula I. Formula II is used for the calculations for within-run duplicates.

$$sd = \sqrt{((\text{sum}x^2 - (\text{sum}x)^2)/n/(n-1))} \dots\dots I$$

$$sd = \sqrt{(\text{sum}d^2/2n)} \dots\dots II$$

where : x = the individual values; n = the number of events
d = the differences between pairs of duplicates

The data is stored in a personal computer using BMB Manager II files. All data manipulations, reports generated etc, are performed using applications written in Manager Math.

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3. Air Emission Samples

Air particulate samples are collected on Hivol filters as well as in Dustfall jars. Both of these media are used to monitor air emissions and each has its own collection and analysis protocols.

3.1 Hivol Filters

Air particulate samples are collected on a variety of filter media. The nominal particulate size fractionation of the Hivol sampler is to exclude airborne particulate greater than 44 μm . Dichotomous samplers take two size fractions, <2.5 μm and 2.5 to 10 μm particulates. The collection media and instrumentation used is determined by the needs of the sampling program.

3.2 Dustfall Jars

Samples are collected in plastic jars lined with polyethylene bags. These sampling devices do not discriminate between particle size or matrix that is collected.

3.3 Both of these sample types may be used to collect samples that can be subjected to microscopy techniques for identification of specific materials in the sample. Currently, QA procedures for this type of analysis consists of comparison of the sample material with material from known sources.

Table 3.1 summarizes the parameters determined, the preparation methods used and the instrument types used for the analysis of air particulates.

TABLE 3.1

Parameter	Collection Device	Preparation	Analysis
Metals	Glass fibre, Delbag, Whatman, Teflon filters Dustfall Jars.	Acid digest	AAS
Anions	" "	Water ext	IC
TSP	Glass fibre and Teflon filters		Gravimetry
Total Dustfall	Dustfall Jar	Filtration	Gravimetry
Carbon	Glass fibre filters	Acid ext	Coulometry, Ignition
Hydride Metals	Glass fibre filters, Dustfall Jars	Acid digest	AAS

3.4 Air Filter Quality Assurance

Air filters, except for Teflon Dichotomous 47 mm filters, are analysed by cutting an aliquot from the filter after TSP determination as required.

Sample duplicates are prepared by cutting a second aliquot from the filter.

Blank filter aliquots are analysed with each analytical run. There are sufficient variations in the filter batches that a blank representing each batch in the analytical run must be analysed.

Between-run composite materials are prepared by grinding and blending exposed glass fibre filters. Selection of filters is made so that two composites of high and low concentrations can be prepared.

Between-run check samples for XRF analysis consist of previously analysed filters.

Table 3.2 indicates the sample descriptors used in the QA summary data, the source and the parameters that they are used to control.

TABLE 3.2

Sample Designation	Type	Parameter
0.3g	Composite filter	AAS Metals
0.3g#2	Composite filter	AAS Metals
cona, conb	high/low filter	XRF lead
conhv1, conhv2	filter aliquot(low)	Uranium
conhv3	filter aliquot (high)	Uranium
.1g	composite filter	Carbon
con1, con2, con3	filter aliquots	Arsenic
.3g-HI, .3g-LO	filter composites	Anions
expose	exposed filters	TSP
nbs	NBS weight	TSP

3.5 Dustfall Quality Assurance

Dustfall samples cannot be subaliquoted to generate sample duplicates. Field sample duplicates were not part of the QA process during 1986.

Dustfall blanks consist of extraction of blank filters of the type used for the preparation of the sample.

Between-run control is maintained by analysis of composite dustfall solutions as well as a dust sample, along with the dustfall samples.

Table 3.3 indicates the sample descriptors used in the QA summary data, the source and the parameters that they are used to control.

TABLE 3.3

Sample Designation	Type	Parameter
100ml comp	Composite solution	AAS Metals
200ml comp	Composite solution	AAS Metals
100-A comp	Composite solution	AAS Metals
200-A comp	Composite solution	AAS Metals
100ml-1,2	Composite solution	AAS Metals
200ml-1,2	Composite solution	AAS Metals
connbs	NBS coal flyash	AAS Metals
.3 Hi,Lo	Composite filter	Anions

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Arsenic
UNIT: Biomaterials

TEST CODE: ASUT

SAMPLE TYPE: Hivol filter
SUPERVISOR: R. Sadana

METHOD CODE: 571AF3

REVISION NO: Original

DATE: January 1983

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-1 cut from a no 11 cork borer having 5.62 cm² area
Container-Manila Envelope
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted->90%
Procedure-Two 1.8cm diameter circles from exposed hivol filters are placed into a graduated test tube and 3 ml of a mixture of HNO₃:H₂SO₄:HClO₄ (6:3:1) is added. Digestion is carried out in a 18x150 mm pyrex graduated test tube at 95 °C set in an aluminum block. It is continued until dense white fumes appear. Add a half ml distilled water and 2 ml of conc. HCl to the cool digestate. Dilute to 15 ml with distilled water and mix well. Feed the digestates to the automated system for the determination of arsenic by the hydride-FAAS technique.

INTERFERENCES: Excessive concentrations of Cu, Fe, and Ni may interfere

REPORTING RESULTS: 2 dec places below 10, 1 below 100, 0 above 100ug/ml
INSTRUMENTATION: Varian atomic absorption spectrophotometer, peristaltic pump, autosampler, open ended quartz tube atomizer, and gas-liquid separator.

Calibration Range: 0-40 ng/ml

Resolution: 0.001 absorbance (unexpanded scale)

Sensitivity: 20 ng/ml = 0.150 Abs. units

Instrument Detection Limit: 0.001 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.001 to .024 ug/M3

Accuracy-99% from EPA water sample

Precision of Controls-

	A	B
mean	.005	.011
std. dev.	.0007	.0013
R.S.D.	14.0%	11.8%

Precision of Duplicates-	low range	mid range	high range
s.d.	0.0010	.00040	.00080
mean	.0020	.0080	.0180

W .5 ng/M3

T 2 ng/M3

CONTROL LIMITS:

REMARKS:

-Accuracy = Ratio of mean to cert. value of ref. std. material X 100

SUMMARY REPORT OF QUALITY CONTROL DATA

ARSENIC IN HIVOL FILTERS

Operating Range = .00100to 0.024 ug/M3

IN - RUN DUPLICATES

Range	<.00100	.00100to0.0048	0.0048to0.0120	0.0120to 0.024	> 0.024
no.	8	66	18	7	9
s.w.		0.00010	0.00040	0.00080	
mean		0.0020	0.0080	0.0180	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
hvc1	27	0.00500	0.00070	14.00
hvc2	26	0.0110	0.00130	11.82
475-3	27	0.0100	0.00159	15.88

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	.00000	.00000

DATE 87/03/12

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Cadmium TEST CODE: CDUT SAMPLE TYPE: Hivol filter
UNIT: Air Quality SUPERVISOR: A.B. Foster

METHOD CODE: 571AA0
REVISION NO: Original DATE: 1971
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-A 19.05x254 mm filter strip
Container-Manila Envelope
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted->90%
Procedure-A 19x254mm sample strip is cut by a scapel and template.
The sample is placed on a brick board and ashed at 500C for 2h. in a
muffle furnace. The strips are transferred to a 100 ml Teflon dishes
and 15ml of a mixture of HF:HNO3:H2O added to each dish. The sample is
taken to dryness by slow evaporation. Five ml of conc. HNO3 are added
and sample is taken to dryness again. One ml of conc. HNO3 is added
followed by 20 ml of water. Sample and contents are allowed to simmer
on a hot plate to reduce volume to 10 ml. The sample is transferred to
a 15 ml calibrated test tube and with washings brought to 15ml mark.
The test tube is placed in an aluminum block and heated for at least 2
hr at 80C. After cooling it is brought to 15 ml mark for AAS analysis.
INTERFERENCES: Radiation buffer is added to standards in order to
minimize effect of hivol matrix on sample.

REPORTING RESULTS: ug/m3, three place after decimal

INSTRUMENTATION: Perkin Elmer model PE5000 atomic absorption spectro-
photometer interfaced with Commodore computer.

Calibration Range: .001-2 ug/ml

Resolution: .001 Abs. Unit

Sensitivity: 1.5ug/ml CD = .200 Abs. Units

Instrument Detection Limit: 0.01 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0003 to 0.037ug/M3

Accuracy-97% from NBS1648 urban particulate

Precision of Controls-

mean	.0273
std. dev.	.00154
R.S.D.	5.64%

B

Precision of Duplicates-low range	mid range	high range
s.d.	.00023	
mean	.0007	

W .5 Ng/M3

T 2.5 Ng/M3

CONTROL LIMITS: A 10% deviation is allowed with the control standard.
With method controls a 20% deviation is allowed before run is rejected
(method controls are >t for control limits to apply).

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM

IN HIVOL FILTERS

Operating Range = .00030to 0.037 ug/m3

IN - RUN DUPLICATES

Range	<.00030	.00030to0.0075	0.0075to0.0187	0.0187to 0.037	> 0.037
no.	284	26	0	0	0
s.w.		0.00023	0.00000	0.00000	
mean		0.0007	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3g	146	0.13164	0.04355	33.08

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
0878	8	.00006	.00006
0749	0	.00000	.00000
0027	2	.00006	.00001
0550	6	.00003	.00003

DATE 87/02/19

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Chromium TEST CODE:CRUT SAMPLE TYPE:Hivol filter
UNIT:Air Quality SUPERVISOR:A.B. Foster

METHOD CODE:571AA0
REVISION NO:Original DATE:1971
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-A 19.05x254 mm filter strip
Container-Manila Envelope
Preservative-
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->90%
Procedure-A 19x254mm sample strip is cut by a scapel and template.
The sample is placed on a brick board and ashed at 500C for 2h. in a
muffle furnace. The strips are transferred to a 100 ml Teflon dishes
and 15ml of a mixture of HF:HNO3:H2O added to each dish. The sample is
taken to dryness by slow evaporation. Five ml of conc. HNO3 are added
and sample is taken to dryness again. One ml of conc. HNO3 is added
followed by 20 ml of water. Sample and contents are allowed to simmer
on a hot plate to reduce volume to 10 ml. The sample is transferred to
a 15 ml calibrated test tube and with washings brought to 15ml mark.
The test tube is placed in an aluminum block and heated for at least 2
hr at 80C. After cooling it is brought to 15 ml mark for AAS analysis.
INTERFERENCES:Radiation buffer is added to standards in order to
minimize effect of hivol matrix on sample.
REPORTING RESULTS:ug/m3, three place after decimal
INSTRUMENTATION:Perkin Elmer model PE5000 atomic absorption spectro-
photometer interfaced with Commodore computer.

Calibration Range:.01-5.00 ug/ml
Resolution:.001 Abs. Unit
Sensitivity: 4.0ug/ml CR = .200 Abs. Units
Instrument Detection Limit:0.13 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0045 to 0.37ug/M3
Accuracy-22.5% from NBS1648 urban particulate
Precision of Controls-

mean	.184
std. dev.	.01504
R.S.D.	8.20%

Precision of Duplicates-low range	mid range	high range
s.d.	.00371	
mean	.0071	

W .005µg/M3

T .025µg/M3

CONTROL LIMITS:A 10% deviation is allowed with the control standard.
With method controls a 20% deviation is allowed before run is rejected
(method controls are >t for control limits to apply).

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CHROMIUM IN HIVOL FILTERS

Operating Range = .00450 to 0.375 ug/m3

IN - RUN DUPLICATES

Range	<.00450	.00450 to 0.0750	0.0750 to 0.1875	0.1875 to 0.375	> 0.375
no.	308	2	0	0	0
S.W.		0.00371	0.00000	0.00000	
mean		0.0071	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3g	156	0.18350	0.01504	8.20

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
0878	215	.01655	.00533
0749	12	.02068	.00550
0027	12	.01520	.01260
0550	74	.02164	.01900

DATE 87/02/19

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Cobalt TEST CODE: COUT SAMPLE TYPE: Hivol filter
UNIT: Air Quality SUPERVISOR: A.B. Foster

METHOD CODE: 571AA0
REVISION NO: Original DATE: 1971
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-A 19.05x254 mm filter strip
Container-Manila Envelope
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted->90%
Procedure-A 19x254mm sample strip is cut by a scapel and template.
The sample is placed on a brick board and ashed at 500C for 2h. in a
muffle furnace. The strips are transferred to a 100 ml Teflon dishes
and 15ml of a mixture of HF:HNO3:H2O added to each dish. The sample is
taken to dryness by slow evaporation. Five ml of conc. HNO3 are added
and sample is taken to dryness again. One ml of conc. HNO3 is added
followed by 20 ml of water. Sample and contents are allowed to simmer
on a hot plate to reduce volume to 10 ml. The sample is transferred to
a 15 ml calibrated test tube and with washings brought to 15ml mark.
The test tube is placed in an aluminum block and heated for at least 2
hr at 80C. After cooling it is brought to 15 ml mark for AAS analysis.
INTERFERENCES: Radiation buffer is added to standards in order to
minimize effect of hivol matrix on sample.

REPORTING RESULTS: ug/m3, two place after decimal

INSTRUMENTATION: Perkin Elmer model PE5000 atomic absorption spectro-
photometer interfaced with Commodore computer.

Calibration Range: .01-5.00 ug/ml
Resolution: .001 Abs. Unit
Sensitivity: 7.0ug/ml CO = .200 Abs. Units
Instrument Detection Limit: 0.04 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.003 to 0.37ug/M3
Accuracy-95.8% from NBS1648 urban particulate
Precision of Controls-

mean	.166
std. dev.	.01161
R.S.D.	6.99%

B

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W .001ug/M3

T .005ug/M3

CONTROL LIMITS: A 10% deviation is allowed with the control standard.
With method controls a 20% deviation is allowed before run is rejected
(method controls are >t for control limits to apply).

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

COBALT IN HIVOL FILTERS

Operating Range = .00300to 0.375 ug/m3

IN - RUN DUPLICATES

Range	<.00300	.00300to0.0750	0.0750to0.1875	0.1875to 0.375	> 0.375
no.	310	0	0	0	0
s.w.		0.00000	0.00000	0.00000	
mean		0.0000	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3g	156	0.16602	0.01161	6.99

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
0878	201	.00239	.00167
0749	13	.00249	.00175
0027	11	.00299	.00357
0550	67	.00188	.00128

DATE 87/02/19

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Copper TEST CODE: CUUT SAMPLE TYPE: Hivol filter
UNIT: Air Quality SUPERVISOR: A.B. Foster

METHOD CODE: 571AA0
REVISION NO: Original DATE: 1971
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-A 19.05x254 mm filter strip
Container-Manila Envelope
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted->90%
Procedure-A 19x254mm sample strip is cut by a scalpel and template.
The sample is placed on a brick board and ashed at 500C for 2h. in a
muffle furnace. The strips are transferred to a 100 ml Teflon dishes
and 15ml of a mixture of HF:HNO3:H2O added to each dish. The sample is
taken to dryness by slow evaporation. Five ml of conc. HNO3 are added
and sample is taken to dryness again. One ml of conc. HNO3 is added
followed by 20 ml of water. Sample and contents are allowed to simmer
on a hot plate to reduce volume to 10 ml. The sample is transferred to
a 15 ml calibrated test tube and with washings brought to 15ml mark.
The test tube is placed in an aluminum block and heated for at least 2
hr at 80C. After cooling it is brought to 15 ml mark for AAS analysis.
INTERFERENCES: Radiation buffer is added to standards in order to
minimize effect of hivol matrix on sample.
REPORTING RESULTS: ug/m3, three place after decimal
INSTRUMENTATION: Perkin Elmer model PE5000 atomic absorption spectro-
photometer interfaced with Commodore computer.

Calibration Range: 0-5 ug/ml
Resolution: .001 Abs. Unit
Sensitivity: 4ug/ml CU = .200 Abs. Units
Instrument Detection Limit: 0.04 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to .35 ug/M3
Accuracy-91% from NBS1648 urban particulate
Precision of Controls-

mean .0904
std. dev. .00627
R.S.D. 6.94

A

B

Precision of Duplicates-low range mid range high range

s.d. .00184 .00285
mean .0129 1.1575

W .005ug/M3

T .020ug/M3

CONTROL LIMITS: A 10% deviation is allowed with the control standard.
With method controls a 20% deviation is allowed before run is rejected
(method controls are >t for control limits to apply).

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER

IN HIVOL FILTERS

Operating Range = .00150to 0.375 ug/m3

IN - RUN DUPLICATES

Range	<.00150	.00150to0.0750	0.0750to0.1875	0.1875to 0.375	> 0.375
no.	3	301	6	0	0
s.w.		0.00184	0.00285	0.00000	
mean		0.0129	1.1575	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3g	154	0.09040	0.00627	6.94

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
0878	203	.00400	.00645
0749	10	.00445	.00167
0027	10	.00351	.00300
0550	72	.00490	.00330

DATE 87/02/19

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Iron
UNIT:Air Quality

TEST CODE:FEUT

SAMPLE TYPE:Hivol filter
SUPERVISOR:A.B. Foster

METHOD CODE:571AAO

REVISION NO:Original

DATE:1971

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-A 19.05x254 mm filter strip
Container-Manila Envelope
Preservative-
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->90%

Procedure-A 19x254mm sample strip is cut by a scapel and template. The sample is placed on a brick board and ashed at 500C for 2h. in a muffle furnace. The strips are transferred to a 100 ml Teflon dishes and 15ml of a mixture of HF:HNO3:H2O added to each dish. The sample is taken to dryness by slow evaporation. Five ml of conc. HNO3 are added and sample is taken to dryness again. One ml of conc. HNO3 is added followed by 20 ml of water. Sample and contents are allowed to simmer on a hot plate to reduce volume to 10 ml. The sample is transferred to a 15 ml calibrated test tube and with washings brought to 15ml mark. The test tube is placed in an aluminum block and heated for at least 2 hr at 80C. After cooling it is brought to 15 ml mark for AAS analysis. INTERFERENCES:Radiation buffer is added to standards in order to minimize effect of hivol matrix on sample.

REPORTING RESULTS:ug/m3, one place after decimal

INSTRUMENTATION:Perkin Elmer model PE5000 atomic absorption spectro-photometer interfaced with Commodore computer.

Calibration Range:.1-100 ug/ml

Resolution:.001 Abs. Unit

Sensitivity: 5.0ug/ml FE = .200 Abs. Units

Instrument Detection Limit:0.2 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.03 to 7.5 ug/M3

Accuracy-94.2% from NBS1648 urban particulate

Precision of Controls-

A

B

mean	2.238
std. dev.	.1601
R.S.D.	7.15%

Precision of Duplicates-low range

mid range

high range

s.d. .00884

mean .1162

W .1 µg/M3

T .5 µg/M3

CONTROL LIMITS:A 10% deviation is allowed with the control standard. With method controls a 20% deviation is allowed before run is rejected (method controls are >t for control limits to apply).

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON

IN HIVOL FILTERS

Operating Range = .03000to 7.500 ug/m3

IN - RUN DUPLICATES

Range	<.03000	.03000to1.5000	1.5000to3.7500	3.7500to 7.500	> 7.500
no.	88	222	0	0	0
s.w.		0.00884	0.00000	0.00000	
mean		0.1162	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3g	156	2.23782	0.16010	7.15

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
0878	217	.53598	.09337
0749	13	.50002	.03325
0027	12	.48749	.02788
0550	74	.51028	.09572

DATE 87/02/19

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Lead
UNIT:Air Quality

TEST CODE:PBUT

SAMPLE TYPE:Hivol filter
SUPERVISOR:A.B. Foster

METHOD CODE:571AAO

REVISION NO:Original

DATE:1971

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-A 19.05x254 mm filter strip
Container-Manila Envelope
Preservative-
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->90%

Procedure-A 19x254mm sample strip is cut by a scapel and template. The sample is placed on a brick board and ashed at 500C for 2h. in a muffle furnace. The strips are transferred to a 100 ml Teflon dishes and 15ml of a mixture of HF:HNO3:H2O added to each dish. The sample is taken to dryness by slow evaporation. Five ml of conc. HNO3 are added and sample is taken to dryness again. One ml of conc. HNO3 is added followed by 20 ml of water. Sample and contents are allowed to simmer on a hot plate to reduce volume to 10 ml. The sample is transferred to a 15 ml calibrated test tube and with washings brought to 15ml mark. The test tube is placed in an aluminum block and heated for at least 2 hr at 80C. After cooling it is brought to 15 ml mark for AAS analysis. INTERFERENCES:Radiation buffer is added to standards in order to minimize effect of hivol matrix on sample.

REPORTING RESULTS:ug/m3, one place after decimal

INSTRUMENTATION:Perkin Elmer model PE5000 atomic absorption spectro-
photometer interfaced with Commodore computer.

Calibration Range:0.1-20.00 ug/ml

Resolution:.001 Abs. Unit

Sensitivity: 20.0ug/ml PB = .200 Abs. Units

Instrument Detection Limit:0.1 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.00375 to 1.50ug/M3

Accuracy-98.9% from NBS1648 urban particulate

Precision of Controls-

A

B

mean	.0161
std. dev.	.0010
R.S.D.	7.55%

Precision of Duplicates-low range

mid range

high range

s.d. .00110

mean .0161

W .01 ug/M3

T .10 ug/M3

CONTROL LIMITS:A 10% deviation is allowed with the control standard. With method controls a 20% deviation is allowed before run is rejected (method controls are >t for control limits to apply).

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD

IN HIVOL FILTERS

Operating Range = .00375to 1.500 ug/m3

IN - RUN DUPLICATES

Range	<.00375	.00375to0.3000	0.3000to0.7500	0.7500to 1.500	> 1.500
no.	37	273	0	0	0
s.w.		0.00110	0.00000	0.00000	
mean		0.0161	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3g	152	0.09722	0.00734	7.55

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
0878	206	.00786	.00601
0749	13	.01259	.00416
0027	12	.01226	.00764
0550	67	.00777	.00440

DATE 87/02/19

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Lead TEST CODE: PBXRF SAMPLE TYPE: Hivol filter
UNIT: Spectroscopy\XRF SUPERVISOR: D. Boomer

METHOD CODE: 509AX2

REVISION NO: 84-1

DATE: Aug 1984

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-A 19.05x254 mm filter strip
Container-Manila Envelope
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-
Procedure-A circular die is used to punch a pair of circles with a 47 mm diameter. They are stored with exposed sides facing each other. The filter is analyzed with a XRF spectrometer employing the following conditions: 50 kV, 40 mA, vacuum on, collimator .15 degrees, crystal 200 LiF, scintillation counter pulse height setting, baseline 1.8, width 4.0, sample holder 34 mm opening. Intensity is measured using an average of two 20 sec counts at the Pb LB line. Counts from unexposed blank filter are subtracted from the measured intensities of the sample to get net intensities. By a stored calibration curve in a computer the Pb/filter is calculated from the net intensities. By dividing by the air volume the ug of Pb/M3 of air is calculated.
INTERFERENCES:

REPORTING RESULTS: ug/m3, one place after decimal
INSTRUMENTATION: Siemens SRS-1 X-ray fluorescence spectrometer, 47mm cutting die, HP9825 computer, Commodore PET computer.

Calibration Range: 0 to 15,000 ug Pb/filter

Resolution:

Sensitivity:

Instrument Detection Limit: 0.1 ug/M3

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.1 to 25 ug/M3

Accuracy-88% (compared to AAS)

Precision of Controls-

	A		B
mean	.412		4.924
std. dev.	.0910		0.130
R.S.D.	22.1%		2.64%
Precision of Duplicates-low range		mid range	high range
s.d.	.103	.496	1.041
mean	.926	9.094	14.139
W .1 ug/M3		T .5 ug/M3	

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD-XRF

IN HIVOL FILTERS

range= 0.100to 25.0 ug/m3

DUPLICATES

range	< 0.100	0.100to 5.00	5.00to 12.50	12.50to 25.0	> 25.0
no.	45	97	18	9	1
s.w.		0.1030	0.4960	1.0410	
mean		0.926	9.094	14.139	

CONTROLS

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
cona	34	0.412	0.0910	22.09
conb	34	4.924	0.1300	2.64

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/09/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Manganese TEST CODE: MNUT SAMPLE TYPE: Hivol filter
UNIT: Air Quality SUPERVISOR: A.B. Foster

METHOD CODE: 571AA0
REVISION NO: Original DATE: 1971
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-A 19.05x254 mm filter strip
Container-Manila Envelope
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted->90%
Procedure-A 19x254mm sample strip is cut by a scapel and template.
The sample is placed on a brick board and ashed at 500C for 2h. in a
muffle furnace. The strips are transferred to a 100 ml Teflon dishes
and 15ml of a mixture of HF:HNO3:H2O added to each dish. The sample is
taken to dryness by slow evaporation. Five ml of conc. HNO3 are added
and sample is taken to dryness again. One ml of conc. HNO3 is added
followed by 20 ml of water. Sample and contents are allowed to simmer
on a hot plate to reduce volume to 10 ml. The sample is transferred to
a 15 ml calibrated test tube and with washings brought to 15ml mark.
The test tube is placed in an aluminum block and heated for at least 2
hr at 80C. After cooling it is brought to 15 ml mark for AAS analysis.
INTERFERENCES: Radiation buffer is added to standards in order to
minimize effect of hivol matrix on sample.

REPORTING RESULTS: ug/m3, two place after decimal

INSTRUMENTATION: Perkin Elmer model PE5000 atomic absorption spectro-
photometer interfaced with Commodore computer.

Calibration Range: 0.1-5 ug/ml

Resolution: .001 Abs. Unit

Sensitivity: 2.5ug/ml MN = .200 Abs. Units

Instrument Detection Limit: 0.03 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.003 to .375 ug/M3

Accuracy-80.3% from NBS1648 urban particulate

Precision of Controls-

mean	.0555
std. dev.	.00477
R.S.D.	8.59%

A

B

Precision of Duplicates-low range

mid range

high range

s.d. .0009

mean .0098

W .005µg/M3

T .025µg/M3

CONTROL LIMITS: A 10% deviation is allowed with the control standard.
With method controls a 20% deviation is allowed before run is rejected
(method controls are >t for control limits to apply).

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN HIVOL FILTERS

Operating Range = .00300to 0.375 ug/m3

IN - RUN DUPLICATES

Range	<.00300	.00300to0.0750	0.0750to0.1875	0.1875to 0.375	> 0.375
no.	216	94	0	0	0
s.w.		0.00090	0.00000	0.00000	
mean		0.0098	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3g	147	0.05549	0.00477	8.59

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
0878	217	.03200	.00379
0749	13	.02890	.00387
0027	12	.02623	.00279
0550	74	.03078	.00515

DATE 87/02/19

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Nickel TEST CODE: NIUT SAMPLE TYPE: Hivol filter
UNIT: Air Quality SUPERVISOR: A.B. Foster

METHOD CODE: 571AA0
REVISION NO: Original DATE: 1971
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-A 19.05x254 mm filter strip
Container-Manila Envelope
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted->90%
Procedure-A 19x254mm sample strip is cut by a scalpel and template. The sample is placed on a brick board and ashed at 500C for 2h. in a muffle furnace. The strips are transferred to a 100 ml Teflon dishes and 15ml of a mixture of HF:HNO3:H2O added to each dish. The sample is taken to dryness by slow evaporation. Five ml of conc. HNO3 are added and sample is taken to dryness again. One ml of conc. HNO3 is added followed by 20 ml of water. Sample and contents are allowed to simmer on a hot plate to reduce volume to 10 ml. The sample is transferred to a 15 ml calibrated test tube and with washings brought to 15ml mark. The test tube is placed in an aluminum block and heated for at least 2 hr at 80C. After cooling it is brought to 15 ml mark for AAS analysis.
INTERFERENCES: Radiation buffer is added to standards in order to minimize effect of hivol matrix on sample.
REPORTING RESULTS: ug/m3, three place after decimal
INSTRUMENTATION: Perkin Elmer model PE5000 atomic absorption spectrophotometer interfaced with Commodore computer.

Calibration Range: 0-5 ug/ml
Resolution: .001 Abs. Unit
Sensitivity: 10ug/ml NI = .200 Abs. Units
Instrument Detection Limit: 0.05 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to .35 ug/M3
Accuracy-106% from NBS1648 urban particulate
Precision of Controls-

mean .1562
std. dev. .01245
R.S.D. 7.97

Precision of Duplicates-low range mid range high range
s.d. .0034
mean .0138

W .005µg/M3

T .020µg/M3

CONTROL LIMITS: A 10% deviation is allowed with the control standard. With method controls a 20% deviation is allowed before run is rejected (method controls are >t for control limits to apply).
REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL

IN HIVOL FILTERS

Operating Range = .00300to 0.375 ug/m3

IN - RUN DUPLICATES

Range	<.00300	.00300to0.0750	0.0750to0.1875	0.1875to 0.375	> 0.375
no.	295	15	0	0	0
s.w.		0.00340	0.00000	0.00000	
mean		0.0138	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3g	155	0.15617	0.01245	7.97

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
0878	137	.00218	.00320
0749	13	.00346	.00166
0027	10	.00377	.00200
0550	70	.00513	.00342

DATE 87/02/19

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Uranium TEST CODE: UUUT SAMPLE TYPE: Hivol Filter
UNIT: Project\Q.C. SUPERVISOR: J. Hipfner

METHOD CODE: 520AE2

REVISION NO:

DATE: 1986

NATURE OF LAST REVISION: Change Fluorometric to ICP/MS

SAMPLE HANDLING:

Quantity Required- 1 cut from no. 11 cork borer with an area of 5.16 cm²
Container-
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.-A % Extracted-
Procedure-A 5.162 sq. cm. circle is cut from a folded glass fibre
hivol filter with a no. 11 cork borer. The filter aliquot is placed in
a test tube graduated at the 10 ml mark and two ml of 8N HNO₃ is added.
The tube and its contents are heated on a hot plate for 2 hrs at 95C.
After cooling the tube is brought to the 10 ml mark with distilled
water. The contents of the tube is shaken and filtered. The filtrate
is analyzed for uranium by an ICP mass spectrometer.

INTERFERENCES:

REPORTING RESULTS: ug of uranium per meter cubed of air
INSTRUMENTATION: Elan 250 ICP mass spectrometer from Sciex, Thorhill
Ont.

Calibration Range: .10 to 5000 ug/L

Resolution: .00001 ug/L

Sensitivity:

Instrument Detection Limit: .1 ug/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- .00005-1.00 ug/M3

Accuracy-

Precision of Controls-

	A	B
mean	.4042	.0051
std. dev.	.03307	.00089
R.S.D.	8.18%	17.7%

Precision of Duplicates-	low range	mid range	high range
s.d.	.00076	.00137	.00118
mean	.0039	.0678	.1052

W .1 ng/M3

T .5 ng/M3

CONTROL LIMITS: Deviation of 15% is allowed with an instrument control
sample. With the method controls 25% deviation from the mean is
allowed before samples would be resubmitted for analysis.

REMARKS: Range of duplicates is lowered over routine operating range
to view how low level samples vary.

SUMMARY REPORT OF QUALITY CONTROL DATA

URANIUM

IN HIVOL FILTERS

Operating Range = .00004to 0.080 ug/m3

IN - RUN DUPLICATES

Range	<.00004	.00004to0.0160	0.0160to0.0400	0.0400to 0.080	> 0.080
no.	0	25	3	1	0
s.w.		0.00076	0.00137	0.00118	
mean		0.0039	0.0678	0.1052	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
conhv4	20	0.40420	0.03307	8.18
conhv5	17	0.0051	0.00089	17.66

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	25	.00008	.00005

DATE 87/04/10

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Vanadium TEST CODE: VVUT SAMPLE TYPE: Hivol filter
UNIT: Air Quality SUPERVISOR: A.B. Foster

METHOD CODE: 571AA0
REVISION NO: Original DATE: 1971
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-A 19.05x254 mm filter strip
Container-Manila Envelope
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted->90%
Procedure-A 19x254mm sample strip is cut by a scapel and template.
The sample is placed on a brick board and ashed at 500C for 2h. in a
muffle furnace. The strips are transferred to a 100 ml Teflon dishes
and 15ml of a mixture of HF:HNO3:H2O added to each dish. The sample is
taken to dryness by slow evaporation. Five ml of conc. HNO3 are added
and sample is taken to dryness again. One ml of conc. HNO3 is added
followed by 20 ml of water. Sample and contents are allowed to simmer
on a hot plate to reduce volume to 10 ml. The sample is transferred to
a 15 ml calibrated test tube and with washings brought to 15ml mark.
The test tube is placed in an aluminum block and heated for at least 2
hr at 80C. After cooling it is brought to 15 ml mark for AAS analysis.
INTERFERENCES: Radiation buffer is added to standards in order to
minimize effect of hivol matrix on sample.
REPORTING RESULTS: ug/m3, two place after decimal
INSTRUMENTATION: Perkin Elmer model PE5000 atomic absorption spectro-
photometer interfaced with Commodore computer.

Calibration Range: 0.1-5 ug/ml
Resolution: .001 Abs. Unit
Sensitivity: 90ug/ml V = .200 Abs. Units
Instrument Detection Limit: 0.1 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0075 to .375 ug/M3
Accuracy-92.5% from NBS1648 urban particulate
Precision of Controls-

mean .04389
std. dev. .00688
R.S.D. 15.68%

B

Precision of Duplicates-low range	mid range	high range
s.d. .00021		
mean .0078		

W .005µg/M3

T .025µg/M3

CONTROL LIMITS: A 10% deviation is allowed with the control standard.
With method controls a 20% deviation is allowed before run is rejected
(method controls are >t for control limits to apply).
REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

VANADIUM IN HIVOL FILTERS

Operating Range = .00750 to 0.375 ug/m3

IN - RUN DUPLICATES

Range	<.00750	.00750 to 0.0750	0.0750 to 0.1875	0.1875 to 0.375	> 0.375
no.	309	1	0	0	0
s.w.		0.00021	0.00000	0.00000	
mean		0.0078	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3g	149	0.04389	0.00688	15.68

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
0878	142	.00524	.00575
0749	9	.00571	.00608
0027	8	.00278	.00276
0550	42	.00279	.00259

DATE 87/02/19

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Zinc TEST CODE: ZNUT SAMPLE TYPE: Hivol filter
UNIT: Air Quality SUPERVISOR: A.B. Foster

METHOD CODE: 571AAO
REVISION NO: Original DATE: 1971
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-A 19.05x254 mm filter strip
Container-Manila Envelope
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted->90%
Procedure-A 19x254mm sample strip is cut by a scapel and template. The sample is placed on a brick board and ashed at 500C for 2h. in a muffle furnace. The strips are transferred to a 100 ml Teflon dishes and 15ml of a mixture of HF:HNO3:H2O added to each dish. The sample is taken to dryness by slow evaporation. Five ml of conc. HNO3 are added and sample is taken to dryness again. One ml of conc. HNO3 is added followed by 20 ml of water. Sample and contents are allowed to simmer on a hot plate to reduce volume to 10 ml. The sample is transferred to a 15 ml calibrated test tube and with washings brought to 15ml mark. The test tube is placed in an aluminum block and heated for at least 2 hr at 80C. After cooling it is brought to 15 ml mark for AAS analysis.
INTERFERENCES: Radiation buffer is added to standards in order to minimize effect of hivol matrix on sample.
REPORTING RESULTS: ug/m3, one place after decimal
INSTRUMENTATION: Perkin Elmer model PE5000 atomic absorption spectro-photometer interfaced with Commodore computer.

Calibration Range: 0-20 ug/ml
Resolution: .001 Abs. Unit
Sensitivity: 1ug/ml ZN = .200 Abs. Units
Instrument Detection Limit: 0.03 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 1.4 ug/M3
Accuracy-96.3% from NBS1648 urban particulate
Precision of Controls-

A

B

mean
std. dev.
R.S.D.

Precision of Duplicates-low range mid range high range
s.d.
mean

W .05 ug/M3

T .25 ug/M3

CONTROL LIMITS: A 10% deviation is allowed with the control standard. With method controls a 20% deviation is allowed before run is rejected (method controls are >t for control limits to apply).
REMARKS: High and variable blank value in filter made measurement of zinc in by glass fibre filter impossible.

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC

IN HIVOL FILTERS

Operating Range = .00150 to 1.500 ug/m3

IN - RUN DUPLICATES

Range	<.00150	.00150 to 0.3000	0.3000 to 0.7500	0.7500 to 1.500	> 1.500
no.	310	0	0	0	0
s.w.		0.00000	0.00000	0.00000	
mean		0.0000	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3g	0	0.00000	0.00000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
0878	0	.00000	.00000
0749	0	.00000	.00000
0027	0	.00000	.00000
0550	0	.00000	.00000

DATE 87/02/19

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Tot. Susp. Part TEST CODE: TSP SAMPLE TYPE: Air
UNIT: Air Quality SUPERVISOR: A.B. Foster

METHOD CODE: 003AB1
REVISION NO: Original DATE: Dec. 83
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-203.2x254.0mm filter & 177.8x228.6mm exposed area
Container-Manila Envelope
Preservative-
Other-Conditioned at 50% humidity

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-
Procedure-Air is drawn through a preweighed glass fibre filter by means of a high-flow blower at such a rate as to allow particulates having diameters of less than 44 μ m to reach the filter. The exposed aerodynamic filter is conditioned at 50% relative humidity and reweighed. The mass concentration of suspended particulate matter is calculated from the filter weight difference and the volume of air sampled.

INTERFERENCES: Oily smog, loss of particulate due to poor adhesion.

REPORTING RESULTS: nearest whole number μ g/ m^3

INSTRUMENTATION: Analytical balance with computer output

Calibration Range: 0.0001-.5g

Resolution: .1 mg

Sensitivity: .1 mg

Instrument Detection Limit: .1 mg

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.05-250 μ g/ M^3

Accuracy-99.9%

Precision of Controls-

	A	B
mean	26.70	31.20
std. dev.	1.55	.90
R.S.D.	5.81%	2.88%

Precision of Duplicates-low range	mid range	high range
s.d.	.585	.405
mean	30.0	82.55

W 1 μ g/ M^3

T 5 μ g/ M^3

CONTROL LIMITS: Two 2g NBS weights are used as a check on instrument calibration. If the weights change below 3.9900 or above 4.000 the balance is sent out for recalibration.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

TSP

IN HIVOL FILTERS

range= 0.050to 250.0 ug/m3

DUPLICATES

range	< 0.050	0.050to 50.00	50.00to125.00	125.00to 250.0	> 250.0
no.	0	39	25	3	0
s.w.		0.5850	0.4050	0.2150	
mean		30.200	82.550	140.900	

CONTROLS

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
cont1	43	26.700	1.5500	5.81
cont2	43	31.200	0.9000	2.88
cont3	57	21.600	1.8000	8.33

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blk1	43	1728.700	0.5350
blk2	43	1755.050	0.8500
blk3	58	1629.750	0.8000

DATE 87/02/26

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Chloride TEST CODE:CLIDUR SAMPLE TYPE:Hivol Filters
UNIT:Air Quality SUPERVISOR:A.B. Foster

METHOD CODE:304A15
REVISION NO:Original DATE:1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-19.05x254mm strip from filter
Container-
Preservative-
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-100 % Extracted-
Procedure-A 19x254mm filter strip is extracted in 50 ml of distilled water at room temperature for 1 hr on a horizontal shaker. The extract is filtered through a serum filter tube and analyzed directly by ion chromatography. Eluent concentration is determined by the condition of the analytical ion exchange column and can vary between 4 to 12 mM KHP. The pH is normally 4.1.

INTERFERENCES:High level of other anions, may interfere due to peak overlap

REPORTING RESULTS:2 dec. places to a max. of 3 significant figures.

INSTRUMENTATION:Single column ion chromatography with conductivity detector, sampler, and sample pump.

Calibration Range:0.1 to 50 mg/L

Resolution:0.05 mg/L

Sensitivity:

Instrument Detection Limit:0.1 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.02 to 20 ug/M3

Accuracy-

Precision of Controls-

	A	B
mean	.374	.554
std. dev.	.1326	.1530
R.S.D.	35.5%	27.6%

Precision of Duplicates-low range mid range high range

s.d.	.0974	.3360
mean	.708	6.554

W .1 ug/M3

T .5 ug/M3

CONTROL LIMITS:For the instrument controls a limit of 15% deviation on the EPA given value is permitted. For the method controls a 25% deviation from the mean is allowed.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CHLORIDE IN HIVOL

range= 0.020to 20.0 ug/m3

DUPLICATES

range	< 0.020	0.020to 4.00	4.00to 10.00	10.00to 20.0	> 20.0
no.	19	37	2	0	0
s.w.		0.0974	0.3360	0.0000	
mean		0.708	6.554	0.000	

CONTROLS

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3-low	34	0.374	0.1326	35.45
.3-high	35	0.554	0.1530	27.62

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blank	78	0.166	0.1132

DATE 87/02/26

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Nitrate TEST CODE: NNOTUR SAMPLE TYPE: Hivol Filters
UNIT: Air Quality SUPERVISOR: A.B. Foster

METHOD CODE: 305AI5
REVISION NO: Original DATE: 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-19.05x254mm filter strip
Container-
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-
Procedure-A 19x254mm filter strip is extracted in 50 ml of distilled water at room temperature for 1 hr on a horizontal shaker. The extract is filtered through a serum filter tube and analyzed directly by ion chromatography. Eluent concentration is determined by the condition of the analytical ion exchange column and can vary between 4 to 12 mM KHP. The pH is normally 4.1.

INTERFERENCES: High level of other anions, may interfere due to peak overlap

REPORTING RESULTS: 2 dec. places to a max. of 3 significant figures.

INSTRUMENTATION: Single column ion chromatography with conductivity detector, sampler, and sample pump.

Calibration Range: 0.5 to 100 mg/L
Resolution: 0.1 mg/L
Sensitivity:
Instrument Detection Limit: 0.5 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.02 to 20 ug/M3

Accuracy-

Precision of Controls-

	A	B
mean	1.376	4.338
std. dev.	.1496	.3140
R.S.D.	10.9%	7.16%

Precision of Duplicates-low range mid range high range

s.d.	.1708	.1810
mean	1.700	5.220

W .1 ug/M3

T .5 ug/M3

CONTROL LIMITS: For the instrument controls a limit of 10% deviation on the EPA given value is permitted. For the method controls a 15% deviation from the mean is allowed.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

NITRATE IN HIVOL

range= 0.020to 20.0 ug/m3

DUPLICATES

range	< 0.020	0.020to 4.00	4.00to 10.00	10.00to 20.0	> 20.0
no.	1	45	12	0	0
s.w.		0.1708	0.1810	0.0000	
mean		1.700	5.220	0.000	

CONTROLS

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3-low	44	1.376	0.1496	10.87
.3-high	46	4.388	0.3140	7.16

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blank	13	0.310	0.0522

DATE 87/02/26

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Sulphate TEST CODE: SSO4UR SAMPLE TYPE: Hivol Filters
UNIT: Air Quality SUPERVISOR: A.B. Foster

METHOD CODE: 305AI5
REVISION NO: Original DATE: 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-19.05x254mm strip
Container-
Preservative-
Other-



SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-

Procedure-A 19x254mm filter strip is extracted in 50 ml of distilled water at room temperature for 1 hr on a horizontal shaker. The extract is filtered through a serum filter tube and analyzed directly by ion chromatography. Eluent concentration is determined by the condition of the analytical ion exchange column and can vary between 4 to 12 mM KHP. The pH is normally 4.1.

INTERFERENCES: High level of other anions, may interfere due to peak overlap

REPORTING RESULTS: 2 dec. places to a max. of 3 significant figures.

INSTRUMENTATION: Single column ion chromatography with conductivity detector, sampler, and sample pump.

Calibration Range: 0.5 to 100 mg/L

Resolution: 0.1 mg/L

Sensitivity:

Instrument Detection Limit: 0.5 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.02 to 20 ug/M3

Accuracy-

Precision of Controls-

	A	B
mean	4.326	12.35
std. dev.	.1646	.8072
R.S.D.	3.80%	6.54%

Precision of Duplicates-low range	mid range	high range
s.d.	.8666	.1622
mean	2.858	5.856
		14.238

W .1 ug/M3

T .5 ug/M3

CONTROL LIMITS: For the instrument controls a limit of 10% deviation on the EPA given value is permitted. For the method controls a 15% deviation from the mean is allowed.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

SULPHATE IN HIVOL

range= 0.020to 20.0 ug/m3

DUPLICATES

range	< 0.020	0.020to 4.00	4.00to 10.00	10.00to 20.0	> 20.0
no.	0	10	41	7	0
s.w.		0.8666	0.1622	0.6074	
mean		2.858	5.856	14.238	

CONTROLS

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3-low	42	4.326	0.1646	3.80
.3-high	46	12.346	0.8072	6.54

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blank	31	0.258	0.2138

DATE 87/02/26

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Carbonate TEST CODE: CCO3UT SAMPLE TYPE: Hivol Filters
UNIT: Air Quality SUPERVISOR: A.B. Foster

METHOD CODE: 590AV1
REVISION NO: Original DATE: 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-3 cuts by no.11 cork borer with an area of 15.5cm²
Container-
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-100 % Extracted-
Procedure-Three 5.16cm² discs are cut from a loaded glass fibre filter, and placed in a sample tube. The tube is inserted on a CO₂ generator. Three ml of 2N HCl containing SnCl₂ is added to the filter. The released CO₂ is swept into the coulometric cell using KOH scrubbed air at a rate of 50 ml/min

INTERFERENCES: None

REPORTING RESULTS: ug of carbonate per meter cubed of air
INSTRUMENTATION: Coulometrics model 5010 coulometer equipped with a 5030 carbonate carbon generator.

Calibration Range: .1 to 3000ug of carbonate carbon

Resolution: 0.1 ug

Sensitivity:

Instrument Detection Limit: 1.0 ug

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.001 to 15 ug/M³

Accuracy-

Precision of Controls-

mean	6.750
std. dev.	.2100
R.S.D.	3.11%

A

B

Precision of Duplicates-low range	mid range	high range
s.d.	.0497	.1516
mean	.484	4.733
		11.048

W .05 ug/M³

T .25 ug/M³

CONTROL LIMITS: For the instrument controls a sample of BaCO₃ is analyzed. A recovery of 99% or greater is expected. For the method controls a deviation of less than 10% of the mean is expected.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CARBONATE IN HIVOL FILTERS

Operating Range = 0.001to 15.0 ug/m3

IN - RUN DUPLICATES

Range	< 0.001	0.001to 3.00	3.00to 7.50	7.50to 15.0	> 15.0
no.	1	60	6	1	0
s.w.		0.0497	0.1516	0.0742	
mean		0.484	4.733	11.048	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qccontrol	68	6.750	0.2100	3.11

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK0025	11	0.324	0.0769
BLK0878	55	0.412	0.0980
BLK0027	2	0.269	0.0202
BLK0550	48	0.325	0.0754

DATE 87/02/27

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Free Carbon TEST CODE:CCELEM SAMPLE TYPE:Hivol Filters
UNIT:Air Quality SUPERVISOR:A.B. Foster

METHOD CODE:005AB1
REVISION NO:Original DATE:1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-1 cut from a no. 15 cork borer (area of 8.3cm²)
Container-
Preservative-
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-100 % Extracted-
Procedure-A no.15 corkborer is used to cut a 8.3 cm² area from a
filter. The filter aliquot is placed in pyrex test tube and acid
digested with conc. HNO₃. After digestion the filter is filtered
through a Gooch crucible with filter disk and layer of alumina. The
Gooch crucible and contents are dried and transferred to combustion
crucible for determination of carbon by a Leco Carbon Determinator.

INTERFERENCES:None

REPORTING RESULTS:ug of free carbon per meter cubed of air
INSTRUMENTATION:Leco WR-112 Carbon Determinator

Calibration Range:1 to 4000 ug carbon

Resolution:1ug of carbon

Sensitivity:1 ug

Instrument Detection Limit:5 ug

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.030 to 60 ug/M³

Accuracy-

Precision of Controls-

	A	B
mean	20.64	
std. dev.	1.53	
R.S.D.	7.44%	

Precision of Duplicates-low range	mid range	high range
s.d. .3816	.8435	.4589
mean 3.541	17.610	46.365

W .1 ug/M³ T .5 ug/M³

CONTROL LIMITS:BaCO₃ is run as an instrument control and 99% recovery
should be obtained. The method controls of duplicates and a .3g ground
up composite should be within 10% of the mean.
REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

FREE CARBON IN HIVOL FILTERS

Operating Range = 0.030to 60.0 ug/m3

IN - RUN DUPLICATES

Range	< 0.030	0.030to 12.00	12.00to 30.00	30.00to 60.0	> 60.0
no.	0	62	5	2	0
s.w.		0.3816	0.8435	0.4589	
mean		3.541	17.610	46.365	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qccontrol	68	20.640	1.5360	7.44

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK0025	13	2.354	0.3726
BLK0878	57	2.185	0.3350
BLK0027	7	2.211	0.2850
BLK0550	53	2.236	0.3537

DATE 87/02/27

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Total Carbon TEST CODE: CCUT SAMPLE TYPE: Hivol Filters
UNIT: Air Quality SUPERVISOR: A.B. Foster

METHOD CODE: 006AB1
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 1 cut with a no. 15 cork borer (area of 8.3 cm²)
Container-
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- 100 % Extracted-
Procedure-A no. 15 corkborer is used to cut a 8.3 cm² area from a filter. The filter aliquot is placed in a combustion crucible and dried at 110C. The filter aliquot is combusted by an induction furnace in an oxygen atmosphere converting all carbon to CO₂. The CO₂ travels into a conductivity cell for measurement of carbon present.

INTERFERENCES: None

REPORTING RESULTS: ug of carbon per meter cubed of air
INSTRUMENTATION: Leco WR-112 Carbon Determinator

Calibration Range: 1 to 4000 ug carbon

Resolution: 1ug of carbon

Sensitivity: 1 ug

Instrument Detection Limit: 5 ug

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.030 to 60 ug/M³

Accuracy-

Precision of Controls-

mean	39.45
std. dev.	1.554
R.S.D.	3.94%

A

B

Precision of Duplicates-low range

s.d. .3187

mean 6.520

mid range

.8595

18.835

high range

.6819

46.950

W .1 ug/M³

T .5 ug/M³

CONTROL LIMITS: BaCO₃ is run as an instrument control and 99% recovery should be obtained. The method controls of duplicates and a .1g ground up composite should be within 10% of the mean.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

TOTAL CARBON IN HIVOL FILTERS

Operating Range = 0.030to 60.0 ug/m3

IN - RUN DUPLICATES

Range	< 0.030	0.030to 12.00	12.00to 30.00	30.00to 60.0	> 60.0
no.	0	43	21	5	0
s.w.		0.3187	0.8595	0.6819	
mean		6.520	18.835	46.950	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qccontrol	69	39.450	1.5540	3.94

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK0025	13	3.023	0.3443
BLK0878	57	2.702	0.3481
BLK0027	7	2.670	0.3040
BLK0550	53	2.647	0.3397

DATE 87/10/15

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Tot. Susp. Part TEST CODE: TSPF, TSPC SAMPLE TYPE: Dichot
UNIT: Air Quality SUPERVISOR: A.B. Foster

METHOD CODE: 003AB1

REVISION NO: Original

DATE: Dec. 83

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- One pair of exposed filters (fine and coarse)

Container- Plastic petrie dish

Preservative-

Other- Conditioned at 50% humidity

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-

Procedure- Air is drawn through a preweighed filters by means of a low-flow blower at such a rate as to allow particulates having diameters of less than 10 μ m to reach the filters. Fine (0 to 2.5 μ m) and coarse (2.5 to 10 μ m) particulates are directed to the filters. The exposed filters are conditioned at 50% relative humidity and reweighed. The mass concentration of suspended particulate matter is calculated from the filter weight difference and the volume of air sampled.

INTERFERENCES: Oily smog, loss of particulate due to poor adhesion.

REPORTING RESULTS: nearest whole number μ g/ m^3

INSTRUMENTATION: Analytical balance with computer output

Calibration Range: 0.0001-.5g

Resolution: .1 mg

Sensitivity: .1 mg

Instrument Detection Limit: .1 mg

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.05-250 μ g/ M^3

Accuracy-99.9%

Precision of Controls-

	A	B
mean	17.7	
std. dev.	0.50	
R.S.D.	2.85%	

Precision of Duplicates-	low range	mid range	high range
s.d.	.34	.90	.217
mean	5.20	13.2	26.8

W 1 μ g/ M^3

T 5 μ g/ M^3

CONTROL LIMITS: Two 2g NBS weights are used as a check on instrument calibration. If the weights change below 3.9900 or above 4.000 the balance is sent out for recalibration.

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

T.S.P. IN DICHOTOMOUS FILTERS

Operating Range = 0.004to 41.7 ug/m3

IN - RUN DUPLICATES

Range	< 0.004	0.004to 8.33	8.33to 20.83	20.83to 41.7	> 41.7
no.	0	21	9	6	0
s.w.		0.3421	0.8987	0.2175	
mean		5.217	13.196	26.779	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
cont1	144	17.717	0.5042	2.85

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Aluminum TEST CODE: ALUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-One pair of exposed filters (fine&coarse fractions)
Container-plastic petri dish
Preservative-None
Other-Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn.- Total Extn.-yes % Extracted-
Procedure-Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode-Mo, Voltage=15KV, Energy Range 0-10KEV, Filter-None, Current=200mA, ROI 1.41-1.55, Analysis Time-200sec.
INTERFERENCES: Br and K (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 39.2 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.12 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0001 to 1.000 µg/M³

Accuracy-103% at 25ug/cm²

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. .0050

.0066

.0222

mean .0939

.2713

.6422

W .005µg/M³

T .025µg/M³

CONTROL LIMITS:

REMARKS:

ALUMINUM IN DICHOTOMOUS FILTERS

IN - RUN DUPLICATES

no.	0	74	22	8	2
s.w.		0.00500	0.00660	0.02220	
mean		0.0939	0.2713	0.6422	

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANK I.D.	NO.	MEAN	STD. DEV.
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3.48

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Arsenic TEST CODE: ASUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1
REVISION NO: 84-1 DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required - One pair of exposed filters (fine & coarse fractions)
Container - plastic petri dish
Preservative - None
Other - Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn. - Total Extn. - yes % Extracted -
Procedure - Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode - Mo, Voltage = 50KV, Energy Range 0-40KEV, Filter - Mo, Current = 200mA, ROI 10.32-10.76, Analysis Time - 100sec.
INTERFERENCES: Pb (corrected by analysis protocol)

REPORTING RESULTS: ug/m³
INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 20.0 ug/cm²
Resolution:
Sensitivity:
Instrument Detection Limit: 0.06 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range - .0001 to 0.050 µg/M³
Accuracy -

Precision of Controls -

A

B

mean
std. dev.
R.S.D.

Precision of Duplicates - low range mid range high range

s.d. .0020 .0036
mean .0027 .0115

W .002 µg/M³

T .010 µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ARSENIC IN DICHOTOMOUS FILTERS

Operating Range = .00010to 0.050 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010to0.0100 0.0100to0.0250 0.0250to 0.050 > 0.050

no.	7	101	2	0	0
s.w.		0.00200	0.00360	0.00000	
mean		0.0027	0.0115	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Barium TEST CODE: BAUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- One pair of exposed filters (fine & coarse fractions)

Container- plastic petri dish

Preservative- None

Other- Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn.- Total Extn.-yes % Extracted-

Procedure- Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode-Mo, Voltage=50KV, Energy Range 0-40KEV, Filter-Mo, Current=200mA, ROI 31.60-32.32, Analysis Time-100sec.

INTERFERENCES: High filter loading (minimized by special background correction)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 55.4 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 1.5 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0001 to 0.50 µg/M³

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. .0606

.1228

mean .0408

.1304

W .05 µg/M³

T .25 µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

BARIUM

IN DICHOTOMOUS FILTERS

Operating Range = .00010 to 0.500 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010 to 0.1000 0.1000 to 0.2500 0.2500 to 0.500 > 0.500

no.	27	61	21	0	1
s.w.		0.06060	0.12280	0.00000	
mean		0.0408	0.1304	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Bromine TEST CODE: BRUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- One pair of exposed filters (fine & coarse fractions)
Container- plastic petri dish
Preservative- None
Other- Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn.- Total Extn.-yes % Extracted-

Procedure- Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode-Mo, Voltage=50KV, Energy Range 0-40KEV, Filter-Mo, Current=200mA, ROI 11.68-12.08, Analysis Time-100sec.
INTERFERENCES: Pb (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 20.7 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.3 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0001 to 0.20 µg/M³

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. .0012

.0015

.0051

mean .0127

.0598

.1535

W .001µg/M³

T .005µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

BROMINE IN DICHOTOMOUS FILTERS

Operating Range = .00010to 0.200 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010to0.0400 0.0400to0.1000 0.1000to 0.200 > 0.200

no.	0	87	20	3	0
s.w.		0.00120	0.00150	0.00510	
mean		0.0127	0.0598	0.1535	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Cadmium TEST CODE: CDUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- One pair of exposed filters (fine & coarse fractions)
Container- plastic petri dish
Preservative- None
Other- Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn.- Total Extn.-yes % Extracted-

Procedure- Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode-Mo, Voltage=50KV, Energy Range 0-40KEV, Filter-Mo, Current=200mA, ROI 22.72-23.36, Analysis Time-100sec.

INTERFERENCES: High filter loading (minimized by special background correction)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 17.8 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.25 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0001 to 0.050 µg/M³

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d.

.0083

.0189

.0198

mean

.0058

.0161

.0299

W .01 µg/M³

T .05 µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM IN DICHOTOMOUS FILTERS

Operating Range = .00010 to 0.050 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010 to 0.0100 0.0100 to 0.0250 0.0250 to 0.050 > 0.050

no.	25	43	35	6	1
s.w.		0.00830	0.01890	0.01980	
mean		0.0058	0.0161	0.0299	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Calcium TEST CODE: CAUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- One pair of exposed filters (fine & coarse fractions)

Container- plastic petri dish

Preservative- None

Other- Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn.- Total Extn.-yes % Extracted-

Procedure- Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode-Mo, Voltage=15KV, Energy Range 0-10KEV, Filter-None, Current=200mA, ROI 3.60-3.80, Analysis Time-200sec.

INTERFERENCES: K (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 21.8 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.03 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- .0001 to 1.50 µg/M³

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates- low range

mid range

high range

s.d. .0085

mean .0569

W .005 µg/M³

T .025 µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CALCIUM IN DICHOTOMOUS FILTERS

Operating Range = .00010to 1.500 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010to0.3000 0.3000to0.7500 0.7500to 1.500 > 1.500

no.	0	109	0	0	1
s.w.		0.00850	0.00000	0.00000	
mean		0.0569	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Chlorine TEST CODE:CLUTD1&2 SAMPLE TYPE:Teflon Filter
UNIT:Spectroscopy/Forensic SUPERVISOR:D.W. Boomer

METHOD CODE:200BX1

REVISION NO:84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-One pair of exposed filters(fine&coarse fractions)

Container-plastic petri dish

Preservative-None

Other-Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-

Procedure-Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are:Anode-W, Voltage=35KV, Energy Range0-20KEV, Filter-Cu, Current=200mA, ROI 2.54-2.70, Analysis Time-200sec.

INTERFERENCES:S and Pb (corrected by analysis protocol)

REPORTING RESULTS:ug/m³

INSTRUMENTATION:EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range:0 to 28.2 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit:0.10 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0001 to 0.500 µg/M³

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. .0061

.0179

.0112

mean .0114

.1339

.3577

W .005µg/M³

T .025µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CHLORINE IN DICHOTOMOUS FILTERS

Operating Range = .00010 to 0.500 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010 to 0.1000 0.1000 to 0.2500 0.2500 to 0.500 > 0.500

no.	47	51	3	1	8
s.w.		0.00610	0.01790	0.01120	
mean		0.0114	0.1339	0.3577	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Chromium TEST CODE:CRUTD1&2 SAMPLE TYPE:Teflon Filter
UNIT:Spectroscopy/Forensic SUPERVISOR:D.W. Boomer

METHOD CODE:200BX1

REVISION NO:84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-One pair of exposed filters(fine&coarse fractions)
Container-plastic petri dish
Preservative-None
Other-Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-

Procedure-Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are:Anode-W, Voltage=35KV, Energy Range0-20KEV, Filter-Cu, Current=200mA, ROI 5.32-5.52, Analysis Time-200sec.
INTERFERENCES: V (corrected by analysis protocol)

REPORTING RESULTS:ug/m³

INSTRUMENTATION:EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range:0 to 16.9 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit:0.02 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0001 to 0.010 µg/M³

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. .0007

.0008

.0012

mean .0007

.0028

.0065

W .5 ng/M³

T 2.5 ng/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CHROMIUM IN DICHOTOMOUS FILTERS

Operating Range = .00010 to 0.010 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010 to 0.0020 0.0020 to 0.0050 0.0050 to 0.010 > 0.010

no.	6	66	28	5	5
s.w.		0.00070	0.00080	0.00120	
mean		0.0007	0.0028	0.0065	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Copper TEST CODE: CUUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required - One pair of exposed filters (fine & coarse fractions)
Container - plastic petri dish
Preservative - None
Other - Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn. - Total Extn. - yes % Extracted -
Procedure - Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode - Mo, Voltage = 50KV, Energy Range 0-40KEV, Filter - Mo, Current = 200mA, ROI 7.88-8.20, Analysis Time - 100sec.
INTERFERENCES: Ni (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 46.8 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.03 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range - 0.0001 to 0.0500 µg/M³

Accuracy -

Precision of Controls -

A

B

mean
std. dev.
R.S.D.

Precision of Duplicates - low range

mid range

high range

s.d. .0021

.0034

.0032

mean .0035

.0145

.0346

W .001 µg/M³

T .005 µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER IN DICHOTOMOUS FILTERS

Operating Range = .00010to 0.050 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010to0.0100 0.0100to0.0250 0.0250to 0.050 > 0.050

no.	9	83	15	3	0
s.w.		0.00210	0.00340	0.00320	
mean		0.0035	0.0145	0.0346	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Iron TEST CODE: FEUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required - One pair of exposed filters (fine & coarse fractions)

Container - plastic petri dish

Preservative - None

Other - Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn. - Total Extn. - yes % Extracted -

Procedure - Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode - Mo, Voltage = 50KV, Energy Range 0-40KEV, Filter - Mo, Current = 200mA, ROI 6.24-6.56, Analysis Time - 100sec.

INTERFERENCES: Mn (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 16.8 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.03 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range - .0001 to 1.00 µg/M³

Accuracy -

Precision of Controls -

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates - low range

mid range

high range

s.d. .0043

.0296

.0072

mean .0615

.2767

.7649

W .005 µg/M³

T .025 µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON

IN DICHOTOMOUS FILTERS

Operating Range = .00010to 1.000 ug/m3

IN - RUN DUPLICATES

Range	<.00010	.00010to0.2000	0.2000to0.5000	0.5000to 1.000	> 1.000
no.	0	95	6	3	6
s.w.		0.00430	0.02960	0.00720	
mean		0.0615	0.2767	0.7649	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Lead TEST CODE: PBUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-One pair of exposed filters (fine & coarse fractions)
Container-plastic petri dish
Preservative-None
Other-Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn.- Total Extn.-yes % Extracted-
Procedure-Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode-Mo, Voltage=50KV, Energy Range 0-40KEV, Filter-Mo, Current=200mA, ROI 12.40-12.80, Analysis Time-100sec.
INTERFERENCES: High filter loading (minimized by special background correction)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 18.2 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.1 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0001 to 1.00 µg/M³

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. .0047

.0078

.0125

mean .0623

.2761

.5521

W .002 µg/M³

T .010 µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD IN DICHOTOMOUS FILTERS

Operating Range = .00010 to 1.000 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010 to 0.2000 0.2000 to 0.5000 0.5000 to 1.000 > 1.000

no.	1	89	18	2	0
s.w.		0.00470	0.00780	0.01250	
mean		0.0623	0.2761	0.5521	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Manganese TEST CODE: MNUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required - One pair of exposed filters (fine & coarse fractions)

Container - plastic petri dish

Preservative - None

Other - Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn. - Total Extn. - yes % Extracted -

Procedure - Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in $\mu\text{g}/\text{cm}^2$. The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode - W, Voltage = 35KV, Energy Range 0-20KEV, Filter - Cu, Current = 200mA, ROI 5.80-6.00, Analysis Time - 200sec.

INTERFERENCES: Cr (corrected by analysis protocol)

REPORTING RESULTS: $\mu\text{g}/\text{m}^3$

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 20.7 $\mu\text{g}/\text{cm}^2$

Resolution:

Sensitivity:

Instrument Detection Limit: 0.01 $\mu\text{g}/\text{cm}^2$

PERFORMANCE CHARACTERISTICS:

Routine Operating Range - 0.0001 to 0.10 $\mu\text{g}/\text{M}^3$

Accuracy -

Precision of Controls -

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates - low range

mid range

high range

s.d. .0007

.0015

.0017

mean .0067

.0293

.0718

W .5 ng/M^3

T 2.5 ng/M^3

CONTROL LIMITS:

REMARKS:

MANGANESE IN DICHOTOMOUS FILTERS

IN - RUN DUPLICATES

no.	4	94	4	4	4
s.w.		0.00070	0.00150	0.00170	
mean		0.0067	0.0293	0.0718	

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANK I.D.	NO.	MEAN	STD. DEV.
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3.70

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Nickel TEST CODE: NIUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- One pair of exposed filters (fine & coarse fractions)

Container- plastic petri dish

Preservative- None

Other- Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn.- Total Extn.-yes % Extracted-
Procedure- Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode-Mo, Voltage=50KV, Energy Range 0-40KEV, Filter-Mo, Current=200mA, ROI 7.32-7.64, Analysis Time-100sec.
INTERFERENCES:

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 18.0 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.05 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0001 to 0.0100 µg/M³

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. .0013

.0024

.0034

mean .0011

.0032

.0065

W .001µg/M³

T .005µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL IN DICHOTOMOUS FILTERS

Operating Range = .00010to 0.010 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010to0.0020 0.0020to0.0050 0.0050to 0.010 > 0.010

no.	17	38	44	8	3
s.w.		0.00130	0.00240	0.00340	
mean		0.0011	0.0032	0.0065	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Phosphorus TEST CODE: PPUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required - One pair of exposed filters (fine & coarse fractions)

Container - plastic petri dish

Preservative - None

Other - Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn. - Total Extn. - yes % Extracted -

Procedure - Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode - Mo, Voltage = 15KV, Energy Range 0-10KEV, Filter - None, Current = 200mA, ROI 1.93-2.09, Analysis Time - 200sec.

INTERFERENCES: Ca and S (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 18.3 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.04 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range - .0001 to 0.200 µg/M³

Accuracy -

Precision of Controls -

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates - low range

mid range

high range

s.d.

.0019

.0057

mean

.0125

.0553

W .001 µg/M³

T .005 µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

PHOSPHORUS IN DICHOTOMOUS FILTERS

Operating Range = .00010 to 0.200 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010 to 0.0400 0.0400 to 0.1000 0.1000 to 0.200 > 0.200

no.	1	100	9	0	0
s.w.		0.00190	0.00570	0.00000	
mean		0.0125	0.0553	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Potassium TEST CODE: KKUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required - One pair of exposed filters (fine & coarse fractions)

Container - plastic petri dish

Preservative - None

Other - Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn. - Total Extn. - yes % Extracted -

Procedure - Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode - Mo, Voltage = 15KV, Energy Range 0-10KEV, Filter - None, Current = 200mA, ROI 3.24-3.38, Analysis Time - 200sec.

INTERFERENCES: Cd and Ca (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 32.2 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.04 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range - .0001 to .50 ug/m³

Accuracy -

Precision of Controls -

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates - low range

mid range

high range

s.d.

.0061

.0052

.0073

mean

.0445

.1469

.3542

W .005µg/M³

T .025µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

POTASSIUM IN DICHOTOMOUS FILTERS

Operating Range = .00010to 0.500 ug/m3

IN - RUN DUPLICATES

Range	<.00010	.00010to0.1000	0.1000to0.2500	0.2500to 0.500	> 0.500
no.	0	81	24	4	1
s.w.		0.00610	0.00520	0.00730	
mean		0.0445	0.1469	0.3542	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Rubidium TEST CODE: RBUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- One pair of exposed filters (fine & coarse fractions)

Container- plastic petri dish

Preservative- None

Other- Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn.- Total Extn.-yes % Extracted-

Procedure- Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode-Mo, Voltage=50KV, Energy Range 0-40KEV, Filter-Mo, Current=200mA, ROI 13.16-13.56, Analysis Time-100sec.

INTERFERENCES: Pb (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 23.2 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.03 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- .0001 to 0.010 µg/M³

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates- low range

mid range

high range

s.d. .0010

.0019

.0032

mean .0008

.0027

.0058

W .001 µg/M³

T .005 µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

RUBIDIUM IN DICHOTOMOUS FILTERS

Operating Range = .00010to 0.010 ug/m3

IN - RUN DUPLICATES

Range	<.00010	.00010to0.0020	0.0020to0.0050	0.0050to 0.010	> 0.010
no.	22	64	21	2	1
s.w.		0.00100	0.00190	0.00320	
mean		0.0008	0.0027	0.0058	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

3.78

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Selenium TEST CODE:SEUTD1&2 SAMPLE TYPE:Teflon Filter
UNIT:Spectroscopy/Forensic SUPERVISOR:D.W. Boomer

METHOD CODE:200BX1

REVISION NO:84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-One pair of exposed filters(fine&coarse fractions)

Container-plastic petri dish

Preservative-None

Other-Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-

Procedure-Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are:Anode-Mo, Voltage=50KV, Energy Range0-40KEV, Filter-Mo, Current=200mA, ROI 11.04-11.24, Analysis Time-100sec.

INTERFERENCES:Pb (corrected by analysis protocol)

REPORTING RESULTS:ug/m³

INSTRUMENTATION:EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range:0 to 53.7 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit:0.02 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0001 to 0.010 µg/M³

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. .0010

.0013

.0020

mean .0009

.0029

.0066

W .001µg/M³

T .005µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

SELENIUM IN DICHOTOMOUS FILTERS

Operating Range = .00010 to 0.010 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010 to 0.0020 0.0020 to 0.0050 0.0050 to 0.010 > 0.010

no.	9	70	19	11	1
s.w.		0.00100	0.00130	0.00200	
mean		0.0009	0.0029	0.0066	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

3.80

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Silicon TEST CODE: SIUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-One pair of exposed filters (fine & coarse fractions)
Container-plastic petri dish
Preservative-None
Other-Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn.- Total Extn.-yes % Extracted-
Procedure-Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode-Mo, Voltage=15KV, Energy Range 0-10KEV, Filter-None, Current=200mA, ROI 1.67-1.81, Analysis Time-200sec. LT
INTERFERENCES: Br and Sr (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 22.2 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.10 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0001 to 1.250 µg/M³

Accuracy-

Precision of Controls-

A

B

mean
std. dev.
R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. .0093

.0092

.0184

mean .1359

.4127

.8476

W .005µg/M³

T .025µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

SILICON IN DICHOTOMOUS FILTERS

Operating Range = .00010to 1.250 ug/m3

IN - RUN DUPLICATES

Range	<.00010	.00010to0.2500	0.2500to0.6250	0.6250to 1.250	> 1.250
no.	1	71	29	8	1
s.w.		0.00930	0.00920	0.01840	
mean		0.1359	0.4127	0.8476	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Strontium TEST CODE: SRUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1
REVISION NO: 84-1 DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required - One pair of exposed filters (fine & coarse fractions)
Container - plastic petri dish
Preservative - None
Other - Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn. - Total Extn. - yes % Extracted -
Procedure - Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode - Mo, Voltage = 50KV, Energy Range 0-40KEV, Filter - Mo, Current = 200mA, ROI 13.92-14.32, Analysis Time - 100sec.
INTERFERENCES: Pb & Br (corrected by analysis protocol)

REPORTING RESULTS: ug/m³
INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 15.5 ug/cm²
Resolution:
Sensitivity:
Instrument Detection Limit: 0.04 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range - 0.0001 to 0.010 µg/M³
Accuracy -

Precision of Controls -

A

B

mean
std. dev.
R.S.D.

Precision of Duplicates - low range

mid range

high range

s.d. .0014

.0025

.0047

mean .0010

.0028

.0062

W .001 µg/M³

T .005 µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

STRONTIUM IN DICHOTOMOUS FILTERS

Operating Range = .00010to 0.010 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010to0.0020 0.0020to0.0050 0.0050to 0.010 > 0.010

no.	36	34	33	6	1
s.w.		0.00140	0.00250	0.00470	
mean		0.0010	0.0028	0.0062	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Sulphur TEST CODE: SSUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required - One pair of exposed filters (fine & coarse fractions)

Container - plastic petri dish

Preservative - None

Other - Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn. - Total Extn. - yes % Extracted -

Procedure - Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode - W, Voltage = 35KV, Energy Range 0-20KEV, Filter - Cu, Current = 200mA, ROI 2.24-2.40, Analysis Time - 200sec.
INTERFERENCES: Pb (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 15.3 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.15 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range - .0001 to 6.000 µg/M³

Accuracy -

Precision of Controls -

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates - low range

mid range

high range

s.d. .1150

.0505

mean .5039

1.8851

W .02 µg/M³

T .10 µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

SULPHUR IN DICHOTOMOUS FILTERS

Operating Range = .00010 to 6.000 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010 to 1.2000 1.2000 to 3.0000 3.0000 to 6.000 > 6.000

no.	0	57	28	21	4
s.w.		0.11500	0.05050	0.09480	
mean		0.5039	1.8851	3.8999	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

3.86

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Titanium TEST CODE: TIUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-One pair of exposed filters (fine & coarse fractions)

Container-plastic petri dish

Preservative-None

Other-Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn.- Total Extn.-yes % Extracted-

Procedure-Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode-W, Voltage=35KV, Energy Range 0-20KEV, Filter-Cu, Current=200mA, ROI 4.42-4.62, Analysis Time-200sec.
INTERFERENCES: Ba (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 14.8 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.03 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0001 to 0.050 µg/M³

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. .0013

.0015

.0055

mean .0041

.0149

.0400

W .001µg/M³

T .005µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

TITANIUM IN DICHOTOMOUS FILTERS

Operating Range = .00010 to 0.050 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010 to 0.0100 0.0100 to 0.0250 0.0250 to 0.050 > 0.050

no.	4	81	20	4	1
s.w.		0.00130	0.00150	0.00550	
mean		0.0041	0.0149	0.0400	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Vanadium TEST CODE: VVUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- One pair of exposed filters (fine & coarse fractions)

Container- plastic petri dish

Preservative- None

Other- Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn.- Total Extn.- yes % Extracted-

Procedure- Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode-W, Voltage=35KV, Energy Range 0-20KEV, Filter-Cu, Current=200mA, ROI 4.42-4.62, Analysis Time-200sec.
INTERFERENCES: Ba & Ti (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 17.9 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.02 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- .0001 to 0.010 µg/M³

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates- low range

mid range

high range

s.d. .0005

.0008

.0010

mean .0008

.0031

.0075

W .5 ng/M³

T 2.5 ng/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

VANADIUM IN DICHOTOMOUS FILTERS

Operating Range = .00010to 0.010 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010to0.0020 0.0020to0.0050 0.0050to 0.010 > 0.010

no.	6	65	28	8	3
s.w.		0.00050	0.00080	0.00100	
mean		0.0008	0.0031	0.0075	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

3.90

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Zinc TEST CODE: ZNUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-One pair of exposed filters (fine & coarse fractions)
Container-plastic petri dish
Preservative-None
Other-Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn.- Total Extn.-yes % Extracted-

Procedure-Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode-Mo, Voltage=50KV, Energy Range 0-40KEV, Filter-Mo, Current=200mA, ROI 8.48-8.80, Analysis Time-100sec.
INTERFERENCES: Ni (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 17.1 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.03 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.0001 to 0.500 µg/M³

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. .0031

.0045

.0098

mean .0241

.1798

.3390

W .002 µg/M³

T .010 µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC IN DICHOTOMOUS FILTERS

Operating Range = .00010 to 0.500 ug/m3

IN - RUN DUPLICATES

Range	<.00010	.00010 to 0.1000	0.1000 to 0.2500	0.2500 to 0.500	> 0.500
no.	0	100	4	2	4
s.w.		0.00310	0.00450	0.00980	
mean		0.0241	0.1798	0.3390	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Zirconium TEST CODE: ZRUTD1&2 SAMPLE TYPE: Teflon Filter
UNIT: Spectroscopy/Forensic SUPERVISOR: D.W. Boomer

METHOD CODE: 200BX1

REVISION NO: 84-1

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required - One pair of exposed filters (fine & coarse fractions)

Container - plastic petri dish

Preservative - None

Other - Samples are placed in plexiglass sample holders when analyzed

SAMPLE PREPARATION: Partial Extn. - Total Extn. - yes % Extracted -

Procedure - Analysis is performed using Energy dispersive x-ray spectrometer. One blank filter and 5 pairs of exposed filters are placed in the instrument sample tray. Background is determined on blank and counts subtracted from sample counts. Concentration is calculated from stored calibration curve in ug/cm². The final concentration is calculated by multiplying by the filter area and dividing by the air volume.

The analysis protocol incorporates a correction factor to compensate for 11% of fine particles being trapped on coarse fraction. Instrument parameters are: Anode - Mo, Voltage = 50KV, Energy Range 0-40KEV, Filter - Mo, Current = 200mA, ROI 15.56-15.96, Analysis Time - 100sec.
INTERFERENCES: Sr, Pb, & Br (corrected by analysis protocol)

REPORTING RESULTS: ug/m³

INSTRUMENTATION: EG&G/Ortec Energy Dispersive X-ray Fluorescence Spectrometer

Calibration Range: 0 to 21.1 ug/cm²

Resolution:

Sensitivity:

Instrument Detection Limit: 0.2 ug/cm²

PERFORMANCE CHARACTERISTICS:

Routine Operating Range - .0001 to 0.050 µg/M³

Accuracy -

Precision of Controls -

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates - low range

mid range

high range

s.d. .0072

.0141

.0001

mean .0052

.0136

.0272

W .005 µg/M³

T .025 µg/M³

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ZIRCONIUM IN DICHOTOMOUS FILTERS

Operating Range = .00010to 0.050 ug/m3

IN - RUN DUPLICATES

Range <.00010 .00010to0.0100 0.0100to0.0250 0.0250to 0.050 > 0.050

no.	24	59	26	1	0
s.w.		0.00720	0.01410	0.00010	
mean		0.0052	0.0136	0.0272	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
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BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/06/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Total Weight TEST CODE: DUSTT SAMPLE TYPE: Dustfall
UNIT: Air Quality SUPERVISOR: Brian Foster

METHOD CODE: 007AB1

REVISION NO: 1

DATE: December, 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-

Container-6" diameter, 12" tall plastic container with plastic liner

Preservative-

Other-Prolonged storage is not recommended because of algal growth

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-97.7

Procedure-The dustfall sample in plastic in ultrasonic bath to loosen particulate matter. After ultrasonification, sample is filtered. The filter is dried and weighed to determine insoluble portion. A 200 ml aliquot of the filtrate is evaporated and weighed to determine the weight of the soluble portion. The two weights are combined to determine the total weight of dustfall.

INTERFERENCES: Insects, tree foliage, bird droppings, algae and fungi are contaminants that could interfere.

REPORTING RESULTS: g/M2/30 days

INSTRUMENTATION: Balance capable of measuring .0001g.

Calibration Range: .002 to 1.0 g.

Resolution: .0001g

Sensitivity: .002g

Instrument Detection Limit: .002g

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.1 to 50 g/M2/30 days

Accuracy-97.7%

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. 0.79

0.90

1.32

mean 2.98

6.99

13.7

W .5

g/M/30 days

CONTROL LIMITS:

REMARKS: Conversion from ug/ml to g/M2/30 days is made with an assumption that a volume of 1000 ml and a exposure time of 30 days.
 $g/M2/30days = Wt \text{ in } g(sol+insol) \times 54.8$

SUMMARY REPORT OF QUALITY CONTROL DATA

TOTAL DUSTFALL IN DUSTFALL

Operating Range = .00010to 20.000 g/m2/30days

IN - RUN DUPLICATES

Range	<.00010	.00010to4.0000	4.0000to10.000	10.000to20.000	>20.000
no.	0	6	8	5	0
s.w.		0.79687	0.90104	1.32476	
mean		2.98330	6.99370	13.69000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ml-1	6	2.87700	0.50416	17.52
200ml-1	5	6.02800	0.46032	7.64
100ml-2	14	0.80008	0.20824	26.03
200ml-2	14	1.60564	0.33976	21.16

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/08/24

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Aluminum TEST CODE: ALUT SAMPLE TYPE: Dustfall
UNIT: Air Quality SUPERVISOR: Brian Foster

METHOD CODE: 532 BAO

REVISION NO: 1

DATE: December, 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation

Container-6" diameter, 12" tall plastic container with plastic liner

Preservative-

Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100

Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in a furnace at 500 C. Then digest with 5 ml of HCl to dryness. Add 3ml of 1:2 HNO₃:H₂O to redissolve sample and make up to 15 ml volume.

Soluble Fraction

Take a 200ml portion of sample, add 5ml of 1:1 HNO₃:H₂O and evaporate to 1 ml. Transfer contents to a test tube and bring to 15 ml volume.

INTERFERENCES:

REPORTING RESULTS: g/M²/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 50.0 ug/ml

Resolution: 0.1 ug/ml

Sensitivity: 45 ug/ml = 0.2 abs. units

Instrument Detection Limit: 0.1 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 0.24 g/M²/30 days

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d.

mean

W 0.001 g/M²/30 days

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM IN DUSTFALL

Operating Range = .00100to 0.2400 g/M2/30 days

IN - RUN DUPLICATES

Range <.00100 .00100to0.0480 0.0480to0.1200 0.1200to0.2400 >0.2400

no.

S.W.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	5	0.0715775	0.0068287	9.54
200ML COMP	5	0.1482559	0.0083137	5.61
100-A COMP	4	0.0035789	0.0015920	44.48
200-A COMP	4	0.0053889	0.0016455	30.53

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK. SOL.	11	0.0009461	0.0005718
BLK. INSOL	9	0.0015632	0.0007651

DATE 87/12/11

ANALYTICAL PROCEDURE

Inorganic Trace Contaminants Section

TEST NAME: Cadmium
UNIT: Air Quality

TEST CODE:CDUT

SAMPLE TYPE:Dustfall

SUPERVISOR: Brian Foster

METHOD CODE:532 BAO

REVISION NO:1

DATE: December, 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation

Container-6" diameter, 12" tall plastic container with plastic liner

Preservative-

Other-

SAMPLE PREPARATION: Partial Extn.- Procedure-Insoluble Fraction	Total Extn.-	% Extracted-100
--	--------------	-----------------

Ash filter in a 50ml beaker in a furnace at 500 C. Then digest with 5 ml of HCl to dryness. Add 3ml of 1:2 HNO₃:H₂O to redissolve sample and make up to 15 ml volume.

Soluble Fraction

Take a 200ml portion of sample, add 5ml of 1:1 HNO₃:H₂O and evaporate to 1 ml. Transfer contents to a test tube and bring to 15 ml volume.

INTERFERENCES:

REPORTING RESULTS: g/M2/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 0.5 ug/ml

Resolution: 0.01 ug/ml

Sensitivity: 1.2 ug/ml = 0.2 abs. units

Instrument Detection Limit: 0.004 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 0.0024 g/M2/30 days

Accuracy-

Precision of Controls-

	A	B
mean	0.00119	0.00242
std. dev.	0.000156	0.000304
R.S.D.	12.9	12.6

	12.5	12.7	12.8
Precision of Duplicates-low range		mid range	high range

s.d.

mean

W 0.00002 g/M/30 days

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM IN DUSTFALL

Operating Range = .00002to 0.0024 g/M2/30 days

IN - RUN DUPLICATES

Range <.00002 .00002to0.0005 0.0005to0.0012 0.0012to0.0024 >0.0024

no.

s.w.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	7	0.0008639	0.0000740	8.57
200ML COMP	7	0.0017277	0.0001358	7.86
100-A COMP	4	0.0023859	0.0000329	1.38
200-A COMP	4	0.0049364	0.0003620	7.33

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK. SOL.	9	0.0000411	0.0000165
BLK. INSOL	9	0.0000411	0.0000206

DATE 87/12/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Calcium TEST CODE: CAUT SAMPLE TYPE: Dustfall
UNIT: Air Quality SUPERVISOR: Brian Foster

METHOD CODE:
REVISION NO: 1 DATE: December, 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation
Container-6" diameter, 12" tall plastic container with plastic liner
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100
Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in furnace at 600 C. Then dissolve with 10 ml of 1:1 HCl:H₂O. Filter sample through a Whatman#4 filter into a 100ml volumetric flask and make to mark. Dilute 1:1 with lanthanum solution.

Soluble Portion

A 10 ml portion of soluble dustfall combined with a equal volume of lanthanum solution is ready for analysis.

INTERFERENCES:

REPORTING RESULTS: g/M²/30 days
INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 10 ug/ml
Resolution: 0.1 ug/ml
Sensitivity: 2.5 ug/ml = 0.2 abs. units
Instrument Detection Limit: 0.05 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 1.21 g/M²/30 days
Accuracy-

Precision of Controls-

	A	B
mean	.153	.186
std. dev.	.037	.150
R.S.D.	24.5	80.4

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W 0.005 g/M²/30 days

CONTROL LIMITS:

REMARKS: Conversion from ug/ml to g/M²/30 days is made with an assumption that the volume of dustfall is 1000 ml
for sol.- g/M²/30 days = (ug/ml x 1000 x 2) x 54.8 / 1,000,000
for insol.- g/M²/30days = (ug/ml x 100 x 2) x 54.8 / 1,000,000

SUMMARY REPORT OF QUALITY CONTROL DATA

CALCIUM IN DUSTFALL

Operating Range = .00500to 1.2100 g/M2/30 days

IN - RUN DUPLICATES

Range <.00500 .00500to0.2420 0.2420to0.6050 0.6050to1.2100 >1.2100

no.

s.w.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	6	3.3537600	0.7578840	22.60
200ML COMP	6	0.1994720	0.0390176	19.56

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK SOL	10	0.0098640	0.0066856
BLK INSOL	10	0.0117272	0.0043292

DATE 87/12/11

3.102

ANALYTICAL PROCEDURE

SUPERVISOR: Brian Foster

SUMMARY REPORT OF QUALITY CONTROL DATA

CHROMIUM IN DUSTFALL

Operating Range = .00050 to 0.0240 g/M2/30 days

IN - RUN DUPLICATES

Range <.00050 .00050 to 0.0048 0.0048 to 0.0120 0.0120 to 0.0240 >0.0240

no.

S.W.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	7	0.0076925	0.0007281	9.47
200ML COMP	6	0.0152205	0.0012300	8.08
100-A COMP	4	0.0083507	0.0007075	8.47
200-A COMP	4	0.0166191	0.0011107	6.68

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK. SOL.	8	0.0002057	0.0001358
BLK. INSOL	7	0.0001645	0.0000617

DATE 87/12/11

3.104

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Cobalt
UNIT: Air Quality

TEST CODE: COUT SAMPLE TYPE: Dustfall
SUPERVISOR: Brian Foster

METHOD CODE: 532 BAO

REVISION NO: 1

DATE: December, 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation
Container-6" diameter, 12" tall plastic container with plastic liner
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100
Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in a furnace at 500 C. Then digest
with 5 ml of HCl to dryness. Add 3ml of 1:2 HNO₃:H₂O to redissolve
sample and make up to 15 ml volume.

Soluble Fraction

Take a 200ml portion of sample, add 5ml of 1:1 HNO₃:H₂O and evaporate
to 1 ml. Transfer contents to a test tube and bring to 15 ml volume.

INTERFERENCES:

REPORTING RESULTS: g/M²/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 5.0 ug/ml

Resolution: 0.1 ug/ml

Sensitivity: 7 ug/ml = 0.2 abs. units

Instrument Detection Limit: 0.04 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 0.024 g/M²/30 days

Accuracy-

Precision of Controls-

	A	B
mean	0.0119	0.0238
std. dev.	0.00136	0.00247
R.S.D.	11.5	10.3

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W 0.0002 g/M/30 days

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

COBALT

IN DUSTFALL

Operating Range = .00020to 0.0240 g/M2/30 days

IN - RUN DUPLICATES

Range <.00020 .00020to0.0048 0.0048to0.0120 0.0120to0.0240 >0.0240

no.

s.w.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	7	0.0082273	0.0007158	8.70
200ML COMP	6	0.0165369	0.0014850	8.98
100-A COMP	4	0.0151382	0.0006870	4.54
200-A COMP	4	0.0299474	0.0007528	2.51

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK. SOL.	8	0.0000411	0.0000329
BLK. INSOL	8	0.0000823	0.0000617

DATE 87/12/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Copper TEST CODE: CUUT SAMPLE TYPE: Dustfall
UNIT: Air Quality SUPERVISOR: Brian Foster

METHOD CODE: 532 BAO

REVISION NO: 1

DATE: December, 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation

Container-6" diameter, 12" tall plastic container with plastic liner

Preservative-

Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100
Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in a furnace at 500 C. Then digest with 5 ml of HCl to dryness. Add 3ml of 1:2 HNO₃:H₂O to redissolve sample and make up to 15 ml volume.

Soluble Fraction

Take a 200ml portion of sample, add 5ml of 1:1 HNO₃:H₂O and evaporate to 1 ml. Transfer contents to a test tube and bring to 15 ml volume.

INTERFERENCES:

REPORTING RESULTS: g/M²/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 5.0 ug/ml

Resolution: 0.1 ug/ml

Sensitivity: 25 ug/ml = 0.2 abs. units

Instrument Detection Limit: 0.02 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 0.024 g/M²/30 days

Accuracy-

Precision of Controls-

	A	B
mean	0.0132	0.0255
std. dev.	0.00173	0.00304
R.S.D.	12.9	11.9

Precision of Duplicates-low range	mid range	high range
s.d.		

W 0.0002 g/M²/30 days

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER

IN DUSTFALL

Operating Range = .00020to 0.0240 g/M2/30 days

IN - RUN DUPLICATES

Range <.00020 .00020to0.0048 0.0048to0.0120 0.0120to0.0240 >0.0240

no.

S.W.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	6	0.0086387	0.0004114	4.76
200ML COMP	5	0.0172773	0.0009461	5.48
100-A COMP	4	0.0094614	0.0005348	5.65
200-A COMP	4	0.0185114	0.0002468	1.33

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK. SOL.	11	0.0004114	0.0000000
BLK. INSOL	12	0.0004114	0.0002057

DATE 87/12/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Iron
UNIT: Air Quality

TEST CODE: FEUT SAMPLE TYPE: Dustfall
SUPERVISOR: Brian Foster

METHOD CODE: 532 BAO

REVISION NO: 1

DATE: December, 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation
Container-6" diameter, 12" tall plastic container with plastic liner
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100
Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in a furnace at 500 C. Then digest
with 5 ml of HCl to dryness. Add 3ml of 1:2 HNO₃:H₂O to redissolve
sample and make up to 15 ml volume.

Soluble Fraction

Take a 200ml portion of sample, add 5ml of 1:1 HNO₃:H₂O and evaporate
to 1 ml. Transfer contents to a test tube and bring to 15 ml volume.

INTERFERENCES:

REPORTING RESULTS: g/M²/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 100 ug/ml

Resolution: 0.1 ug/ml

Sensitivity: 6.0 ug/ml = 0.2 abs. units

Instrument Detection Limit: 0.1 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 0.48 g/M²/30 days

Accuracy-

Precision of Controls-

	A	B
mean	0.345	0.594
std. dev.	0.163	0.202
R.S.D.	41.3	34.1

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W 0.0005 g/M/30 days

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON

IN DUSTFALL

Operating Range = .00050to 0.4800 g/M2/30 days

IN - RUN DUPLICATES

Range <.00050 .00050to0.0960 0.0960to0.2400 0.2400to0.4800 >0.4800

no.

s.w.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	7	0.0796279	0.0059841	7.52
200ML COMP	5	0.1625714	0.0080105	4.93
100-A COMP	4	0.0441189	0.0047899	10.86
200-A COMP	4	0.0873122	0.0064115	7.34

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK. SOL.	11	0.0013822	0.0004015
BLK. INSOL	9	0.0038956	0.0008581

DATE 87/12/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Lead
UNIT: Air Quality

TEST CODE: PBUT

SAMPLE TYPE: Dustfall

SUPERVISOR: Brian Foster

METHOD CODE: 532 BAO

REVISION NO: 1

DATE: December, 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation

Container-6" diameter, 12" tall plastic container with plastic liner

Preservative-

Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100
Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in a furnace at 500 C. Then digest with 5 ml of HCl to dryness. Add 3ml of 1:2 HNO₃:H₂O to redissolve sample and make up to 15 ml volume.

Soluble Fraction

Take a 200ml portion of sample, add 5ml of 1:1 HNO₃:H₂O and evaporate to 1 ml. Transfer contents to a test tube and bring to 15 ml volume.

INTERFERENCES:

REPORTING RESULTS: g/M²/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 20 ug/ml

Resolution: 0.1 ug/ml

Sensitivity: 25 ug/ml = 0.2 abs. units

Instrument Detection Limit: 0.06 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 0.096 g/M²/30 days

Accuracy-

Precision of Controls-

	A	B
mean	0.0501	0.0978
std. dev.	0.00645	0.01007
R.S.D.	12.8	10.3

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W 0.0005 g/M/30 days

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD

IN DUSTFALL

Operating Range = .00050to 0.0960 g/M2/30 days

IN - RUN DUPLICATES

Range <.00050 .00050to0.0192 0.0192to0.0480 0.0480to0.0960 >0.0960

no.

s.w.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	7	0.0295360	0.0014521	4.92
200ML COMP	7	0.0592777	0.0040067	6.76
100-A COMP	4	0.0114359	0.0005759	5.04
200-A COMP	4	0.0214321	0.0002427	1.13

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK. SOL.	9	0.0009050	0.0005718
BLK. INSOL	12	0.0005348	0.0002180

DATE 87/12/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Magnesium TEST CODE: MGUT SAMPLE TYPE: Dustfall
UNIT: Air Quality SUPERVISOR: Brian Foster

METHOD CODE:
REVISION NO: 1 DATE: December, 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation
Container-6" diameter, 12" tall plastic container with plastic liner
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100
Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in furnace at 600 C. Then dissolve with 10 ml of 1:1 HCl:H₂O. Filter sample through a Whatman#4 filter into a 100ml volumetric flask and make to mark. Dilute 1:1 with lanthanum solution.

Soluble Portion

A 10 ml portion of soluble dustfall combined with a equal volume of lanthanum solution is ready for analysis.

INTERFERENCES:

REPORTING RESULTS: g/M2/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 2.0 ug/ml

Resolution: 0.1 ug/ml

Sensitivity: 0.3 ug/ml = 0.2 abs. units

Instrument Detection Limit: 0.02 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to .241 g/M2/30 days

Accuracy-

Precision of Controls-

	A	B
mean	.0193	.0229
std. dev.	.0057	.0203
R.S.D.	29.4	88.4

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W 0.002 g/M/30 days

CONTROL LIMITS:

REMARKS: Conversion from ug/ml to g/M2/30 days is made with an assumption that the volume of dustfall is 1000 ml
for sol.- g/M2/30 days = (ug/ml x 1000 x 2) x 54.8 / 1,000,000
for insol.- g/M2/30days = (ug/ml x 100 x 2) x 54.8 / 1,000,000

SUMMARY REPORT OF QUALITY CONTROL DATA

MAGNESIUM IN DUSTFALL

Operating Range = .00200to 0.2410 g/M2/30 days

IN - RUN DUPLICATES

Range <.00200 .00200to0.0482 0.0482to0.1205 0.1205to0.2410 >0.2410

no.

s.w.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	6	0.3288000	0.0197280	6.00
200ML COMP	6	0.0197280	0.0012056	6.11

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK SOL	6	0.0010960	0.0004384
BLK INSOL	10	0.0005480	0.0002411

DATE 87/12/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Manganese TEST CODE: MNUT SAMPLE TYPE: Dustfall
UNIT: Air Quality SUPERVISOR: Brian Foster

METHOD CODE: 532 BAO

REVISION NO: 1

DATE: December, 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation

Container-6" diameter, 12" tall plastic container with plastic liner

Preservative-

Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100
Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in a furnace at 500 C. Then digest with 5 ml of HCl to dryness. Add 3ml of 1:2 HNO₃:H₂O to redissolve sample and make up to 15 ml volume.

Soluble Fraction

Take a 200ml portion of sample, add 5ml of 1:1 HNO₃:H₂O and evaporate to 1 ml. Transfer contents to a test tube and bring to 15 ml volume.

INTERFERENCES:

REPORTING RESULTS: g/M²/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 5.0 ug/ml

Resolution: 0.1 ug/ml

Sensitivity: 2.5 ug/ml = 0.2 abs. units

Instrument Detection Limit: 0.04 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 0.024 g/M²/30 days

Accuracy-

Precision of Controls-

	A	B
mean	0.0144	0.0292
std. dev.	0.00206	0.00411
R.S.D.	14.5	14.1

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W 0.0002 g/M/30 days

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN DUSTFALL

Operating Range = .00020 to 0.0240 g/M2/30 days

IN - RUN DUPLICATES

Range <.00020 .00020 to 0.0048 0.0048 to 0.0120 0.0120 to 0.0240 >0.0240

no.

s.w.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	7	0.0075691	0.0006458	8.53
200ML COMP	6	0.0152205	0.0011025	7.24
100-A COMP	4	0.0014809	0.0000864	5.83
200-A COMP	4	0.0028384	0.0001028	3.62

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK. SOL.	12	0.0000823	0.0000370
BLK. INSOL	12	0.0001645	0.0000699

DATE 87/12/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Molybdenum TEST CODE: MOUT SAMPLE TYPE: Dustfall
UNIT: Air Quality SUPERVISOR: Brian Foster

METHOD CODE: 532 BAO

REVISION NO: 1

DATE: December, 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation
Container-6" diameter, 12" tall plastic container with plastic liner
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100
Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in a furnace at 500 C. Then digest with 5 ml of HCl to dryness. Add 3ml of 1:2 HNO₃:H₂O to redissolve sample and make up to 15 ml volume.

Soluble Fraction

Take a 200ml portion of sample, add 5ml of 1:1 HNO₃:H₂O and evaporate to 1 ml. Transfer contents to a test tube and bring to 15 ml volume.

INTERFERENCES:

REPORTING RESULTS: g/M²/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 5.0 ug/ml

Resolution: 0.1 ug/ml

Sensitivity: 25 ug/ml = 0.2 abs. units

Instrument Detection Limit: 0.04 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 0.024 g/M²/30 days

Accuracy-

Precision of Controls-

	A	B
mean	0.0095	0.0017
std. dev.	0.00267	0.00497
R.S.D.	27.8	29.5

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W 0.0002 g/M²/30 days

CONTROL LIMITS:

REMARKS: To convert from ug/ml to g/M²/30 days an assumption is made that the dustfall contains 1000 ml of solution

For sol.- g/M²/30 days = (ug/ml x 15 x 1000/200) x 54.8/ 1,000,000

For insol- g/M²/30 days = (ug/ml x 15) x 54.8/1,000,000

SUMMARY REPORT OF QUALITY CONTROL DATA

MOLYBDENUM IN DUSTFALL

Operating Range = .00020 to 0.0240 g/M2/30 days

IN - RUN DUPLICATES

Range <.00020 .00020 to 0.0048 0.0048 to 0.0120 0.0120 to 0.0240 >0.0240

no.

S.W.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	7	0.0076925	0.0011189	14.55
200ML COMP	6	0.0154673	0.0015591	10.08
100-A COMP	4	0.0003291	0.0002098	63.75
200-A COMP	4	0.0003291	0.0001933	58.75

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK. SOL.	10	0.0001645	0.0001028
BLK. INSOL	10	0.0002057	0.0001687

DATE 87/12/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Nickel
UNIT: Air Quality

TEST CODE: NIUT SAMPLE TYPE: Dustfall
SUPERVISOR: Brian Foster

METHOD CODE: 532 BAO

REVISION NO: 1

DATE: December, 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation
Container-6" diameter, 12" tall plastic container with plastic liner
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100
Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in a furnace at 500 C. Then digest
with 5 ml of HCl to dryness. Add 3ml of 1:2 HNO₃:H₂O to redissolve
sample and make up to 15 ml volume.

Soluble Fraction

Take a 200ml portion of sample, add 5ml of 1:1 HNO₃:H₂O and evaporate
to 1 ml. Transfer contents to a test tube and bring to 15 ml volume.

INTERFERENCES:

REPORTING RESULTS: g/M²/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 5.0 ug/ml

Resolution: 0.1 ug/ml

Sensitivity: 7 ug/ml = 0.2 abs. units

Instrument Detection Limit: 0.04 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 0.024 g/M²/30 days

Accuracy-

Precision of Controls-

	A	B
mean	0.0119	0.0238
std. dev.	0.00132	0.00189
R.S.D.	11.1	8.0

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W 0.0002 g/M/30 days

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL

IN DUSTFALL

Operating Range = .00020to 0.0240 g/M2/30 days

IN - RUN DUPLICATES

Range <.00020 .00020to0.0048 0.0048to0.0120 0.0120to0.0240 >0.0240

no.

S.W.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	7	0.0078982	0.0006458	8.18
200ML COMP	6	0.0163723	0.0008968	5.48
100-A COMP	4	0.0070343	0.0002098	2.98
200-A COMP	4	0.0137807	0.0002221	1.61

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK. SOL.	10	0.0002468	0.0001728
BLK. INSOL	11	0.0001645	0.0000699

DATE 87/12/11

3.120

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Potassium TEST CODE: KKUT SAMPLE TYPE: Dustfall
UNIT: Air Quality SUPERVISOR: Brian Foster

METHOD CODE:
REVISION NO: 1 DATE: December, 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation
Container-6" diameter, 12" tall plastic container with plastic liner
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100
Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in furnace at 600 C. Then dissolve with 10 ml of 1:1 HCl:H₂O. Filter sample through a Whatman#4 filter into a 100ml volumetric flask and make to mark. Dilute 1:1 with lanthanum solution.

Soluble Portion

A 10 ml portion of soluble dustfall combined with a equal volume of lanthanum solution is ready for analysis.

INTERFERENCES:

REPORTING RESULTS: g/M2/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 10.0 ug/ml
Resolution: 0.01 ug/ml
Sensitivity: 1.8 ug/ml = 0.2 abs. units
Instrument Detection Limit: 0.5 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 1.21 g/M2/30 days

Accuracy-

Precision of Controls-

	A	B
mean	.0133	.0157
std. dev.	.0036	.0092
R.S.D.	27.3	58.5

Precision of Duplicates-low range mid range high range

s.d.

mean

W 0.05 g/M/30 days

CONTROL LIMITS:

REMARKS: Conversion from ug/ml to g/M2/30 days is made with an assumption that the volume of dustfall is 1000 ml
for sol.- g/M2/30 days = (ug/ml x 1000 x 2) x 54.8 / 1,000,000
for insol.- g/M2/30 days = (ug/ml x 100 x 2) x 54.8 / 1,000,000

SUMMARY REPORT OF QUALITY CONTROL DATA

POTASSIUM IN DUSTFALL

Operating Range = .05000to 1.2100 g/M2/30 days

IN - RUN DUPLICATES

Range <.05000 .05000to0.2420 0.2420to0.6050 0.6050to1.2100 >1.2100

no.

S.W.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	5	0.4438800	0.0723360	16.30
200ML COMP	5	0.0274000	0.0053704	19.60

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK SOL	4	0.0032880	0.0028496
BLK INSOL	9	0.0008768	0.0009316

DATE 87/12/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Sodium
UNIT: Air Quality

TEST CODE: NAUT SAMPLE TYPE: Dustfall
SUPERVISOR: Brian Foster

METHOD CODE:

REVISION NO: 1

DATE: December, 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation
Container-6" diameter, 12" tall plastic container with plastic liner
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100
Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in furnace at 600 C. Then dissolve with 10 ml of 1:1 HCl:H₂O. Filter sample through a Whatman#4 filter into a 100ml volumetric flask and make to mark. Dilute 1:1 with lanthanum solution.

Soluble Portion

A 10 ml portion of soluble dustfall combined with a equal volume of lanthanum solution is ready for analysis.

INTERFERENCES:

REPORTING RESULTS: g/M²/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 1.0 ug/ml

Resolution: 0.001 ug/ml

Sensitivity: 0.7 ug/ml = 0.2 abs. units

Instrument Detection Limit: 0.05 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to .121 g/M²/30 days

Accuracy-

Precision of Controls-

	A	B
mean	.0229	.0229
std. dev.	.0181	.0186
R.S.D.	83.2	81.1

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W 0.005 g/M²/30 days

CONTROL LIMITS:

REMARKS: Conversion from ug/ml to g/M²/30 days is made with an assumption that the volume of dustfall is 1000 ml
for sol.- g/M²/30 days = (ug/ml x 1000 x 2) x 54.8 / 1,000,000
for insol.- g/M²/30 days = (ug/ml x 100 x 2) x 54.8 / 1,000,000

SUMMARY REPORT OF QUALITY CONTROL DATA

SODIUM

IN DUSTFALL

Operating Range = .00500to 0.1210 g/M2/30 days

IN - RUN DUPLICATES

Range <.00500 .00500to0.0242 0.0242to0.0605 0.0605to0.1210 >0.1210

no.

s.w.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	4	11.0641200	1.7081160	15.44
200ML COMP	6	0.7650080	0.1591392	20.80

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK SOL	7	0.0109600	0.0038360
BLK INSOL	5	0.0187416	0.0032003

DATE 87/12/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Vanadium TEST CODE: VVUT SAMPLE TYPE: Dustfall
UNIT: Air Quality SUPERVISOR: Brian Foster

METHOD CODE: 532 BAO

REVISION NO: 1

DATE: December, 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation
Container-6" diameter, 12" tall plastic container with plastic liner
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100
Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in a furnace at 500 C. Then digest
with 5 ml of HCl to dryness. Add 3ml of 1:2 HNO₃:H₂O to redissolve
sample and make up to 15 ml volume.

Soluble Fraction

Take a 200ml portion of sample, add 5ml of 1:1 HNO₃:H₂O and evaporate
to 1 ml. Transfer contents to a test tube and bring to 15 ml volume.

INTERFERENCES:

REPORTING RESULTS: g/M²/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 5.0 ug/ml

Resolution: 0.1 ug/ml

Sensitivity: 75 ug/ml = 0.2 abs. units

Instrument Detection Limit: 0.08 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 0.024 g/M²/30 days

Accuracy-

Precision of Controls-

	A	B
mean	0.0119	0.0243
std. dev.	0.00140	0.00267
R.S.D.	11.9	11.0

Precision of Duplicates-low range mid range high range

s.d.

mean

W 0.0005 g/M²/30 days

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

VANADIUM IN DUSTFALL

Operating Range = .00050to 0.0240 g/M2/30 days

IN - RUN DUPLICATES

Range <.00050 .00050to0.0048 0.0048to0.0120 0.0120to0.0240 >0.0240

no.

s.w.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	0	0.0000000	0.0000000	0.00
200ML COMP	2	0.0004114	0.0000000	0.00
100-A COMP	4	0.0078159	0.0005759	7.37
200-A COMP	4	0.0156319	0.0003291	2.11

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK. SOL.	4	0.0004114	0.0000000
BLK. INSOL	4	0.0004114	0.0002057

DATE 87/12/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Zinc
UNIT: Air Quality

TEST CODE: ZNUT SAMPLE TYPE: Dustfall
SUPERVISOR: Brian Foster

METHOD CODE: 532 BAO

REVISION NO: 1

DATE: December, 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation
Container-6" diameter, 12" tall plastic container with plastic liner
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100
Procedure-Insoluble Fraction

Ash filter in a 50ml beaker in a furnace at 500 C. Then digest
with 5 ml of HCl to dryness. Add 3ml of 1:2 HNO₃:H₂O to redissolve
sample and make up to 15 ml volume.

Soluble Fraction

Take a 200ml portion of sample, add 5ml of 1:1 HNO₃:H₂O and evaporate
to 1 ml. Transfer contents to a test tube and bring to 15 ml volume.

INTERFERENCES:

REPORTING RESULTS: g/M²/30 days

INSTRUMENTATION: A P.E. 5000 atomic absorption spectrophotometer

Calibration Range: 0 to 20 ug/ml

Resolution: 0.1 ug/ml

Sensitivity: 0.8 ug/ml = 0.2 abs. units

Instrument Detection Limit: 0.02 ug/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 0.096 g/M²/30 days

Accuracy-

Precision of Controls-

	A	B
mean	0.0563	0.111
std. dev.	0.00949	0.0163
R.S.D.	16.9	14.7

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W 0.0002 g/M²/30 days

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC

IN DUSTFALL

Operating Range = .00020 to 0.0960 g/M2/30 days

IN - RUN DUPLICATES

Range <.00020 .00020 to 0.0192 0.0192 to 0.0480 0.0480 to 0.0960 >0.0960

no.

s.w.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
100ML COMP	7	0.0369406	0.0033485	9.06
200ML COMP	5	0.0765962	0.0015755	2.06
100-A COMP	4	0.0342667	0.0035377	10.32
200-A COMP	4	0.0618282	0.0022625	3.66

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK. SOL.	12	0.0006170	0.0002345
BLK. INSOL	12	0.0005348	0.0003867

DATE 87/12/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Nitrate TEST CODE: NNOTUR SAMPLE TYPE: Dustfall
UNIT: Air Quality SUPERVISOR: Brian Foster

METHOD CODE: 601AI5

REVISION NO: 1

DATE: 1983

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-20ml

Container-6" diameter, 12" tall plastic container with plastic liner

Preservative-None

Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-

Procedure-An aliquot of the soluble portion of the dustfall sample is taken for anion analysis by ion chromatography. Eluent concentration is determined by the condition of the analytical ion exchange column and can vary between 4 and 12 mM KHP. The pH is normally 4.1.

INTERFERENCES: High levels of other anions may interfere due to peak overlap.

REPORTING RESULTS: g/M2/30 days

INSTRUMENTATION: Single column ion chromatograph with conductivity cell detector, sampler, and sample pump.

Calibration Range: 0.5 to 100 mg/L

Resolution: 0.1 mg/L

Sensitivity:

Instrument Detection Limit: 0.5 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.03 to 5.5 g/M2/30 days

Accuracy-

Precision of Controls-

	A	B
mean	.377	1.202
std. dev.	.0410	.0861
R.S.D.	10.9%	7.16%

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W 0.1 g/M/30 days

CONTROL LIMITS: For instrument controls a limit of 10% deviation on the EPA given value is permitted.

REMARKS: Conversion from ug/ml to g/M2/30 days is made with an assumption that the volume of dustfall is 1000 ml
for sol.- g/M2/30 days = (ug/ml x 1000) x 54.8 / 1,000,000

SUMMARY REPORT OF QUALITY CONTROL DATA

NITRATE IN DUSTFALL

Operating Range = .05000to 5.500 g/m2/30days

IN - RUN DUPLICATES

Range <.05000 .05000to1.1000 1.1000to 2.750 2.750to 5.500 > 5.500

no.

s.w.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3-low	44	0.37700	0.04099	10.87
.3-high	46	1.20230	0.08610	7.16

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blank	13	.08494	.01431

DATE 87/08/24

3.132

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Sulphate TEST CODE: SSO4UR SAMPLE TYPE: Dustfall
UNIT: Air Quality SUPERVISOR: Brian Foster

METHOD CODE: 601AI5

REVISION NO: 1

DATE: 1983

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-20ml

Container-6" diameter, 12" tall plastic container with plastic liner

Preservative-None

Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-

Procedure-An aliquot of the soluble portion of the dustfall sample is taken for anion analysis by ion chromatography. Eluent concentration is determined by the condition of the analytical ion exchange column and can vary between 4 and 12 mM KHP. The pH is normally 4.1.

INTERFERENCES: High levels of other anions may interfere due to peak overlap.

REPORTING RESULTS: g/M2/30 days

INSTRUMENTATION: Single column ion chromatograph with conductivity cell detector, sampler, and sample pump.

Calibration Range: 0.5 to 100 mg/L

Resolution: 0.1 mg/L

Sensitivity:

Instrument Detection Limit: 0.5 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.03 to 5.5 g/M2/30 days

Accuracy-

Precision of Controls-

	A	B
mean	1.186	3.382
std. dev.	.0451	.2212
R.S.D.	3.80%	6.54%

Precision of Duplicates-low range mid range high range

s.d.

mean

W 0.1 g/M/30 days

CONTROL LIMITS: For instrument controls a limit of 10% deviation on the EPA given value is permitted.

REMARKS: Conversion from ug/ml to g/M2/30 days is made with an assumption that the volume of dustfall is 1000 ml
for sol.- g/M2/30 days = (ug/ml x 1000) x 54.8 / 1,000,000

SUMMARY REPORT OF QUALITY CONTROL DATA

SULPHATE IN DUSTFALL

Operating Range = .05000to 5.500 g/m2/30days

IN - RUN DUPLICATES

Range <.05000 .05000to1.1000 1.1000to 2.750 2.750to 5.500 > 5.500

no.

s.w.

mean

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
.3-low	42	1.18550	0.04510	3.80
.3-high	46	3.38300	0.22120	6.54

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blank	31	.07070	.05860

DATE 87/08/24

3.134

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Uranium TEST CODE: UUUT SAMPLE TYPE: Dustfall
UNIT: Project/QC SUPERVISOR: Jerry Hipfner

METHOD CODE:

REVISION NO: 2

DATE: February, 1986

NATURE OF LAST REVISION: detection system -fluorometric to ICP/MS

SAMPLE HANDLING:

Quantity Required-200ml to 1500 ml precipitation

Container-6" diameter, 12" tall plastic container with plastic liner

Preservative-

Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100

Procedure-After a total evaporation of dustfall sample in a 250 ml beaker, 15ml of 5.3N HNO₃ is added to the sample. The beaker is covered with a watch glass, and heated at 95C for 15 minutes. The sample is cooled and filtered. The HNO₃ soluble portion or filtrate is brought to 50 ml and is ready for analysis.

The filter is further digested by a HF-NO₃ digestion procedure and brought to a final volume of 10 ml. .25g of nbs coal fly ash sample is digested along with the HNO₃ insoluble dustfall samples.

INTERFERENCES:

REPORTING RESULTS: g/M2/30 days

INSTRUMENTATION: A Sciex Elan 250 ICP/MS mass spectrometer

Calibration Range: 0 to 1.0 mg/L

Resolution: usually 0.00001 mg/L

Sensitivity: .100mg/L is between 35000 and 85000 counts per second

Instrument Detection Limit: 0.0001 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 - .0027 mg/M2/days

Accuracy-

Precision of Controls-

	A	B
mean	.0006227	
std. dev.	.0001082	
R.S.D.	17.4%	

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W 0.00001 g/M/30 days

CONTROL LIMITS: 15% on instrument control before recalibration

25% on nbs sample before rejecting run

single sample analysis does not allow repeats

REMARKS: Conversion from mg/L to g/M2/30 days

$$\text{g/M2/30 days} = \{[(\text{mg/L HNO}_3 \text{ sol.} \times 50) + (\text{mg/L HNO}_3 \text{ insol.} \times 10)] \times 54.8\} / 10^6$$

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Fluoride TEST CODE: FFIDUR SAMPLE TYPE: Dustfall
UNIT: Air Quality SUPERVISOR: Brian Foster

METHOD CODE: 605AIE
REVISION NO: 1 DATE: 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-20ml
Container-6" diameter, 12" tall plastic container with plastic liner
Preservative-None
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-
Procedure-A 10 ml aliquot of the soluble portion of the dustfall is taken for fluoride analysis by ion selective electrode. To the 10ml portion, 40 ml of TISAB buffer is added and the amount of fluoride calculated against a standard curve made the same day.

INTERFERENCES: Complexing metal interferences are removed by addition of TISAB complexing buffer.
REPORTING RESULTS: g/M2/30 days to 2 decimal places, and max. of 3 s.f.
INSTRUMENTATION: Orion Fluoride ISE used with Technicon automated ISE module or manually operated Orion 901 microprocessor.

Calibration Range: 0 to 1 mg/L
Resolution: 0.01 mg/L
Sensitivity:
Instrument Detection Limit: 0.01 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.003 to .274 g/M2/30 days

Accuracy-

Precision of Controls-

A

B

mean
std. dev.
R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d.

mean

W 0.01 g/M/30 days

CONTROL LIMITS: For instrument controls a limit of 10% deviation on the EPA given value is permitted.

REMARKS: Conversion from ug/ml to g/M2/30 days is made with an assumption that the volume of dustfall is 1000 ml
for sol.- g/M2/30 days = (ug/ml x 1000 x 5) x 54.8 / 1,000,000



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MOE/ANN/ITC/ALRL

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